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FOREWORD

The *Proceedings* of the American Society for Testing Materials are published annually and include all reports and papers offered to the Society during the current year and accepted by the Administrative Committee on Papers and Publications for the *Proceedings*, together with discussion.

The table of contents and subject and author indexes cover all papers and reports published by the Society during the current year, which in addition to those appearing in the *Proceedings* include those accepted for publication in the ASTM BULLETIN or in Special Technical Publications.

A list of the Special Technical Publications published by the Society in 1956 is given on page 1498 of this volume. This supplements the lists appearing in the *Proceedings* from 1948 to 1955 covering all special publications published by the Society up to and including 1955.

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OF THE

FIFTY-NINTH ANNUAL MEETING

ATLANTIC CITY, N. J., JUNE 18–22, 1956

This summary of the Fifty-ninth Annual Meeting of the American Society for Testing Materials, held at the Chalfonte-Haddon Hall Hotel, Atlantic City, N. J., June 18–22, 1956, is a record of the transactions of the meeting, including the actions taken on the various recommendations submitted by the technical committees. In all, 32 technical sessions were held.

The registered attendance of the meeting is as follows: Members present or represented, 1497; committee members, 887; guests, 512; total, 2896; ladies, 204.

The Proceedings are set forth session by session. There were 72 reports and 108 formal papers presented. The record with respect to each has been briefed, the recommendations in the reports have been grouped so as to cover the acceptance of material for publication as tentative, such as new specifications, methods of test, revisions of tentatives, and proposed revisions of existing standards, and, as a separate group, the approval of matters that were referred to letter ballot of the Society, comprising the adoption of tentatives as standard and the adoption of revisions of standards. Accordingly, wherever the action is reported "adopted as standard" or "adopted as standard, revisions in," it is understood that this indicates approval of the Annual Meeting for reference to letter ballot of the Society.[1] The various recommendations so recorded are included in the Society letter ballot. The actions designated as "accepted as tentative" or "accepted as tentative, revisions in" are self-evident as indicating acceptance by the Society at the Annual Meeting for publication as tentative. Designations that have since been assigned to new tentatives are included as information.

All papers, unless otherwise noted, are published in the Proceedings.

While all the items on the program are recorded under the particular session in which they are presented, for convenience in locating actions with respect to any particular report, the accompanying list is presented of all reports together with the page reference where the actions thereon are recorded.

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[1] The letter ballot on recommendations affecting standards, distributed to the Society membership, will be canvassed on September 10, 1956; see Editorial Note, p. 31.

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FIRST SESSION—OPENING SESSION

MONDAY, JUNE 18, 2:00 P.M.

FORMAL OPENING OF THE FIFTY-NINTH ANNUAL MEETING, PRESIDENT C. H. FELLOWS

The session was first formally opened by President C. H. Fellows, who called attention to the very complete program of events that were to follow during the ensuing week. The meeting was then turned over to the session chairman, H. T. Williams, Duquesne Slag Products Co.

SYMPOSIUM ON SPECIFIC GRAVITY OF BITUMINOUS COATED AGGREGATES[2]

SESSION CHAIRMAN: H. T. WILLIAMS

Introduction—The Role of Specific Gravity in the Design and Control of Bituminous Paving Mixtures—C. A. Carpenter, presented by the author.

Measurement of Bituminous Concrete Core Densities and Density of Bituminous Coated Aggregates by Means of a Volumeter—F. M. Williams, L. E. Grimmer, M. M. McAdams, and S. Yanoshek, presented by Mr. Williams.

Measurement of Maximum Theoretical Specific Gravity of a Bituminous Mixture by Solvent Immersion—Paul J. Serafin, presented by the author.

Development and Application of the Effective Specific Gravity of Bituminous Coated Aggregates—J. Rogers Martin and Andrew H. Layman, Jr., presented by Mr. Martin.

Maximum Specific Gravity of Bituminous Mixtures by Vacuum Saturation Procedure—James M. Rice, presented by the author.

Specific Gravity and Voids Relationships in Bituminous Pavement Mix Design—J. L. McRae, presented by the author.

(*Continued in the Thirteenth and Sixteenth Sessions*)

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[2] Issued as separate publication *ASTM STP No. 191.*

SECOND SESSION—METALS

MONDAY, JUNE 18, 8:00 P.M.
SESSION CHAIRMAN: G. V. SMITH

Impact Properties of Quenched-and-Tempered Alloy Steels—J. P. Sheehan and Harry Schwartzbart, presented in abstract by the Chairman G. V. Smith.

A Tensile Study of Brittle Behavior of a Rimmed Steel in the Temperature Range of −269 to 160 C—E. T. Wessel, presented by the author.

Effects of a Number of Heat Treating and Testing Variables on the Notch Strength of 4340 Steel—George Sachs, Volker Weiss, and Eugene Klier, presented by Mr. Weiss.

An Empirical Relationship Between Rupture Life and Minimum Creep Rate in Creep Rupture Tests—Forest C. Monkman and Nicholas I. Grant, presented by Mr. Monkman.

The Effects of Strain Rate and Temperature on the Stress-Strain Relations of Deep-Drawing Steel—R. J. MacDonald, R. L. Carlson, and W. T. Lankford, presented by Mr. MacDonald.

THIRD SESSION—CONCRETE

MONDAY, JUNE 18, 8:00 P.M.
SESSION CHAIRMAN: A. A. BATES

Effect of Revibration on the Properties of Portland-Cement Concrete—D. H. Sawyer and S. F. Lee, presented by Mr. Sawyer.

A Statistical Study of Flexural Strength of Concrete Beams—William C. Otto, presented by Mr. L. A. Palmer.

Automatic Equipment and Cooperative Test Results for the Four ASTM Freezing-and-Thawing Methods for Concrete—H. T. Arni, B. E. Foster, and R. A. Clevenger, presented by Mr. Arni.

Correlation of Sonic Properties of Concrete with Creep and Relaxation—Tien S. Chang and Clyde E. Kesler, presented by Mr. Kesler.

Use of Fly Ash in Concrete—Albert G. Timms and William E. Grieb, presented by Mr. Grieb.

Studies Relating to the Testing of Fly Ash for Use in Concrete—R. H. Brink and W. J. Halstead, presented by Mr. Brink.

FOURTH SESSION—SYMPOSIUM ON ION-EXCHANGE AND CHROMATOGRAPHY IN ANALYTICAL CHEMISTRY[3]

MONDAY, JUNE 18, 8:00 P.M.
SESSION CHAIRMAN: H. F. BEEGHLY

Ion-Exchange in Analytical Chemistry—Robert Kunin, presented by the author.

The Use of Ion-Exchange Techniques in Analytical Chemistry and Radiochemistry—J. E. Hudgens, presented by the author.

Anion-Exchange in Metal Separations—Kurt Kraus, presented by the author.

Ion-Exchange in the Analysis of Metals—John L. Hague, presented by the author.

FIFTH SESSION—SYMPOSIUM ON SOLDER[4]

TUESDAY, JUNE 19, 9:30 A.M.
HONORARY CHAIRMAN: PAST-PRESIDENT G. H. CLAMER
SESSION CHAIRMAN: G. O. HIERS

Introduction by Bruce W. Gonser.

Dip Soldering Printed Circuits—E. Miller, presented by the author.

Controlling Quality on Soldered Electrical Connections—W. H. Rombach, presented by the author.

Solders, Fluxes and Techniques for Soldering Aluminum—J. D. Dowd, presented by the author.

Corrosive Fluxes—Their Role in Soldering—R. M. MacIntosh, presented by the author.

(*Symposium Continued in the Ninth, Fourteenth, and Fifteenth Sessions*)

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[3] Issued as separate publication *ASTM STP No. 195.*

[4] Issued as separate publication *ASTM STP No. 189.*

SIXTH SESSION—SYMPOSIUM ON pH MEASUREMENT[5]

TUESDAY, JUNE 19, 9:30 A.M.

SESSION CHAIRMAN: R. G. BATES

Introduction by E. B. Ashcraft.

Meaning and Standardization of pH Measurements—Roger G. Bates, presented by the author.

Performance Studies of Reference Electrodes and Their Components at High Temperatures and Pressures—John E. Leonard, presented by the author.

Modern Developments in pH Instrumentation—W. R. Clark and G. A. Perley, presented by Mr. Clark.

(*Symposium Continued in the Tenth Session*)

SEVENTH SESSION—COMMITTEE REPORT SESSION

TUESDAY, JUNE 19, 11:30 A.M.

SESSION CHAIRMAN: R. R. LITEHISER

Committee C-3 on Chemical-Resistant Mortars:

Report presented by Beaumont Thomas, chairman, and the following action taken:

Adopted as Standard:

Test for Bond Strength of Chemical Resistant Mortars (C 321 – 54 T)

Committee C-8 on Refractories:

Report presented in the absence of the chairman by R. E. Hess, and the following actions taken:

Accepted as Tentative:

Methods for Chemical Analysis of Refractory Materials (C 18 – 56 T)

Accepted as Tentative, Revisions in:

Methods of Chemical Analysis of Refractory Materials (C 18 – 52)

Def. of Terms Relating to Refractories (C 71 – 55)

Adopted as Standard, Revisions in:

Test for Pyrometric Cone Equivalent (P.C.E.) of Refractory Materials (C 24 – 46)

Test for Disintegration of Fireclay Refractories in an Atmosphere of Carbon Monoxide (C 288 – 54)

Classification of Fireclay Refractories (C 27 – 41)

Committee E-5 on Fire Tests of Materials and Construction:

Report presented by R. C. Corson, secretary, and the following actions taken:

Accepted as Tentative, Revisions in:

Methods of Fire Tests of Door Assemblies (E 152 – 55 T)

Methods of Fire Tests of Roof Coverings (E 108 – 55T)

Methods of Fire Tests of Building Construction and Materials (E 119 – 55)

The report as preprinted called for the addition of a new Section 26 to Methods E 119, whereas this was intended as a replacement of the present Section 26.

Committee E-6 on Methods of Testing Building Constructions:

Report presented in the absence of the chairman by E. C. Shuman, and accepted as a report of progress.

Paper:

The Effect of Weathering on Certain Mortars Exposed in Brick Masonry With and Without Caps and Flashings (Informal Presentation)—J. W. McBurney, presented by the author.

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[5] Issued as separate publication *ASTM STP No. 190.*

EIGHTH SESSION—LUNCHEON, PRESENTATION OF AWARDS OF MERIT, INTRODUCTION OF NEW OFFICERS, PRESIDENT'S ADDRESS, AWARD OF HONORARY MEMBERSHIPS, REPORT OF BOARD OF DIRECTORS, RECOGNITION OF 40- AND 50-YEAR MEMBERS

TUESDAY, JUNE 19, 12:00 NOON

CHAIRMAN: PAST-PRESIDENT N. L. MOCHEL

About 370 were present at this annual luncheon session, including a number of ladies and visitors.

Awards of Merit:

The Chairman of the 1956 Award of Merit Committee, G. L. Werley, read brief citations and presented the following to President Fellows, who conferred on them, on behalf of the Board of Directors, the certificate of the Award of Merit:

L. Drew Betz
Harry A. Bright
Simon Collier
William L. Fink
Harry F. Gonnerman
Bruce W. Gonser
Fred Hubbard
Roland P. Koehring
Vincent T. Malcolm
John W. McBurney
Harry R. Wolf

Election of Officers:

Results of the letter ballot on election of new officers were announced by L. Drew Betz on behalf of the Teller's Committee consisting of L. Drew Betz, chairman, and L. P. Mains. The results were as follows:

For President, to serve for one year:
  R. A. Schatzel, 1796 votes.
For Vice-President, to serve for two years:
  K. B. Woods, 1800 votes.
For Directors, to serve for three years:
  M. N. Clair, 1786 votes.
  H. C. Cross, 1786 votes.
  G. H. Harnden, 1779 votes.
  R. R. Litehiser, 1797 votes.
  C. R. Stock, 1781 votes.

The newly-elected members of the Board of Directors who were present were introduced, as were President-elect Schatzel and Vice-President Woods, the latter two men responding briefly.

Presidential Address:

The annual President's Address was presented by C. H. Fellows. This address is printed in the July, 1956, ASTM BULLETIN.

Award of Honorary Memberships:

The Executive Secretary read citations for the following four members who had been elected by the Board of Directors to Honorary Membership in the Society:

H. J. Gilkey
J. T. MacKenzie
H. B. Oatley
A. E. White

Mr. Fellows presented the certificate to Messrs. Gilkey, MacKenzie, and Oatley. Mr. T. A. Boyd received the certificate for Mr. White.

Report of the Board of Directors:

R. J. Painter, Executive Secretary, presented a brief résumé of the Report of the Board of Directors.

Recognition of 50- and 40-Year Members, Special Presentations:

Chairman Mochel read the names of the thirteen members, both individual and company, who had been continuously affiliated with the Society for 50 years. Certificates to that effect were presented. The procedure was repeated for thirty-seven 40-year members. A complete list of the 50- and 40-year members appears in the July, 1956, ASTM BULLETIN.

The Chairman then presented the Past-President's pin to retiring President C. H. Fellows. Mention was made that this meeting brings to a close for H. L. Maxwell eight years on the Board of Directors, as Director, Vice-President, President, and Past-President. Recognition was also given to N. A. Fowler, T. F. Olt, and J. R. Townsend as retiring directors.

NINTH SESSION—SYMPOSIUM ON SOLDER[4]

(*Symposium Continued from Fifth Session*)

TUESDAY, JUNE 19, 2:30 P.M.

SESSION CHAIRMAN: G. H. HARNDEN

Tin Disease in Solder Type Alloys—A. Bornemann, presented by the author.

Gray Tin Formation in Soldered Joints Stored at Low Temperature—W. L. Williams, presented by the author.

The Performance of Some Soft Solders at Elevated Temperatures and Pressures—R. M. Evans and H. E. Pattee, presented by Mr. Pattee.

Fluxes and Their Application in Soldering—A. Z. Mample, presented by the author.

(*Symposium Continued in the Fourteenth and Fifteenth Sessions*)

TENTH SESSION—SYMPOSIUM ON pH MEASUREMENT[5]

(*Symposium Continued from Sixth Session*)

TUESDAY, JUNE 19, 2:30 P.M.

SESSION CHAIRMAN: ROBERT BURNS

Problems in Measurement of the pH of Blood and Other Biological Fluids—Julius Sendroy, Jr., presented by the author.

Quantitative Applications of pH Measurements in Analytical Chemistry—Henry Freiser, presented by the author.

Theoretical and Practical Problems in the Measurement of Acidity in Nonaqueous Media—Martin Kilpatrick, presented by the author.

Indicator Acidity Functions for Nonaqueous and Mixed Solvents—M. A. Paul and F. A. Long, presented by Mr. Paul.

ROUND-TABLE DISCUSSION ON STATISTICAL ASPECTS OF ASTM SPECIFICATIONS

TUESDAY, JUNE 19, 2:30 P.M.

Under auspices of Committee E-11 on Quality Control

H. F. Dodge, Chairman

O. P. Beckwith, Secretary

Sponsored by Subcommittee I on ASTM Problems

C. A. Bicking, Chairman

This round-table session was arranged in order to provide an opportunity for members of the Society and particularly of the technical committees to discuss their mutual problems in the field of statistical analysis and quality control techniques. To initiate the discussion the following three papers were presented:

Comment on Applying Statistical Requirements in ASTM Standards—presented by Frank Grubbs.

Acceptance Provisions in Some ASTM Specifications and Their Relationship to Buyer-Seller Relations—presented by W. R. Pabst, Jr.

Analysis of Quality Control Provisions in ASTM Specifications and Suggestions for Improvement—presented by Oliver Beckwith.

DISCUSSION ON NON-COMMERCIAL MICROSCOPIC ACCESSORIES

TUESDAY, JUNE 19, 2:30 P.M.

Sponsored by Subcommittee 28 on Microscopy of Committee E-1 on Methods of Testing

F. G. Foster, Chairman

A. F. Kirkpatrick, Secretary

J. H. Hamill, Chairman of Discussion Session

All microscopists have made some accessories for the optical or electron microscopes, or for the preparation of samples; however, they have had little opportunity for the general exchange of information on such accessories. In accordance with the purposes of Subcommittee 28 on Microscopy, of Committee E-1 on Methods of Testing, to develop standards and methods applicable in industrial microscopy and to sponsor symposiums and collect and distribute microscopical information, this session on accessories was organized.

To stimulate discussion from the floor, several brief presentations were scheduled.

ELEVENTH SESSION—COMMITTEE REPORT SESSION

TUESDAY, JUNE 19, 4:30 P.M.

SESSION CHAIRMAN: R. C. ALDEN

Committee A-1 on Steel:

Report presented in the absence of the chairman by W. F. Collins, vice-chairman, and the following actions taken:

Accepted as Tentative:

Spec. for High Tensile Strength Carbon-Manganese Steel Plates for Unfired Pressure Vessels (A 397 – 56 T)

Spec. for Chromium-Silicon Steel Spring Wire (A 401 – 56 T)

Spec. for Chromium-Silicon Steel Valve Spring Quality Wire (A 402 – 56 T)

Spec. for Factory-Made Wrought Austenitic Steel Welding Fittings (A 403 – 56 T), with the deletion in footnote *a* of Table I of the words "if the welding overlay is removed."

Spec. for Forged or Rolled Alloy-Steel Pipe Flanges, Forged Fittings, and Valves and Parts Specially Heat-Treated for High-Temperature Service (A 404 – 56 T)

Spec. for Seamless Ferritic Alloy-Steel Pipe Specially Heat-Treated for High-Temperature Service (A 405 – 56 T)

Rec. Practice for the Selection of Steel Bar Compositions According to Section (A 400 – 56 T)

Accepted as Tentative, Revisions in:

Spec. for General Requirements for Delivery of Rolled Steel Plates, Shapes, Sheet Piling, and Bars for Structural Use (A 6 – 54 T)

Spec. for Steel for Bridges and Buildings (A 7 – 55 T)

Spec. for Low-Carbon Steel Track Bolts and Nuts (A 76 – 55)

Spec. for Seamless Steel Boiler Tubes (A 83 – 55 T)

Spec. for Electric-Fusion-Welded Steel Pipe for High-Temperature Service (A 155 – 55 T)

Spec. for Electric-Resistance-Welded Steel and Open-Hearth Iron Boiler Tubes (A 178 – 55 T)

Spec. for Seamless Cold-Drawn Low-Carbon Steel Heat-Exchanger and Condenser Tubes (A 179 – 55 T)

Spec. for Forged or Rolled Alloy-Steel Pipe Flanges, Forged Fittings, and Valves and Parts for High-Temperature Service (A 182 – 55 T), with the withdrawal from the report as preprinted of the revision of Table II.

Spec. for Alloy-Steel Bolting Materials for High-Temperature Service (A 193 – 55 T)

Spec. for Carbon and Alloy-Steel Nuts for Bolts for High-Pressure and High-Temperature Service (A 194 – 55 T)

Spec. for Seamless Cold-Drawn Intermediate Alloy-Steel Heat-Exchanger and Condenser Tubes (A 199 – 55 T)

Spec. for Seamless Alloy-Steel Boiler, Superheater, and Heat-Exchanger Tubes (A 213 – 55 T), with the withdrawal from the report as preprinted of new grades TP 304 L and TP 316 L.

Spec. for Electric-Resistance-Welded Steel Heat-Exchanger and Condenser Tubes (A 214 – 55 T)
Spec. for Carbon-Steel Castings Suitable for Fusion Welding for High-Temperature Service (A 216 – 53 T)
Spec. for Electric-Resistance-Welded Steel Boiler and Superheater Tubes for High-Pressure Service (A 226 – 55 T)
Spec. for Factory-Made Wrought Carbon Steel and Ferritic Alloy-Steel Welding Fittings (A 234 – 52a T)
Spec. for Welded Austenitic Stainless Steel Boiler, Superheater, Heat-Exchanger, and Condenser Tubes (A 249 – 55 T), with the withdrawal from the report as preprinted of the new grades TP 304 L and TP 316 L.
Spec. for Seamless and Welded Austenitic Stainless Steel Pipe (A 312 – 55 T), with the withdrawal from the report as preprinted of new grades TP 304 L and TP 316 L.
Spec. for Alloy-Steel Bolting Materials for Low-Temperature Service (A 320 – 55 T)
Spec. for Heavy-Walled Carbon and Low Alloy-Steel Castings for Steam Turbines (A 356 – 52 T)
Spec. for 5 Per Cent Chromium, 0.5 Per Cent Molybdenum Steel Plates for Boilers and Other Pressure Vessels (A 357 – 54 T)
Spec. for Electric-Fusion-Welded Austenitic Chromium-Nickel Alloy-Steel Pipe for High-Temperature Service (A 358 – 55 T)
Spec. for Structural Steel for Welding (A 373 – 54 T)
Spec. for Seamless Austenitic Steel Pipe for High-Temperature Central-Station Service (A 376 – 55 T)
Spec. for Chromium-Molybdenum Steel Plates for Boilers and Other Pressure Vessels (A 387 – 55 T)
Spec. for Alloy-Steel Castings Normalized and Drawn for High-Pressure and Elevated Temperature Service (A 389 – 55 T)
Spec. for Alloy-Steel Chain (A 391 – 55 T)

The committee withdrew from the report as preprinted the revisions of the Specification for Untreated Carbon-Steel Axles for Export and for General Industrial Use (A 383 – 55 T).

Adopted as Standard:

Spec. for Open-Hearth Carbon-Steel Rails (A 1 – 54 T)
Spec. for General Requirements for Delivery of Rolled Steel Plates of Flange and Firebox Qualities (A 20 – 54 T)
Spec. for Boiler and Firebox Steel for Locomotives (A 30 – 54 T)
Spec. for Open-Hearth Iron Plates of Flange Quality (A 129 – 54 T)
Spec. for Chromium-Manganese-Silicon (CMS) Alloy-Steel Plates for Boilers and Other Pressure Vessels (A 202 – 54 T)
Spec. for Nickel-Steel Plates for Boilers and Other Pressure Vessels (A 203 – 54 T)
Spec. for Molybdenum-Steel Plates for Boilers and Other Pressure Vessels (A 204 – 54 T)
Spec. for Manganese-Vanadium Steel Plates for Boilers and Other Pressure Vessels (A 225 – 54 T)
Spec. for High-Tensile Strength Carbon-Manganese-Silicon Steel Plates for Boilers and Other Pressure Vessels (A 299 – 54 T)
Spec. for Steel Plates for Pressure Vessels for Service at Low Temperatures (A 300 – 54a T)
Spec. for Manganese-Molybdenum Steel Plates for Boilers and Other Pressure Vessels (A 302 – 54 T)
Spec. for Low-Carbon High-Nickel Steel Plate for Pressure Vessels (A 353 – 54 T)
Method of Tension Testing of Steel Spring Wire (A 318 – 48 T)
Spec. for Nickel-Chromium-Molybdenum Steel Bars for Springs (A 332 – 51 T)

Adopted as Standard, Revisions in:

Spec. for Oil-Tempered Steel Spring Wire (A 299 – 41)
Spec. for Steel Tires (A 26 – 39)
Spec. for Structural Steel for Locomotives and Cars (A 113 – 54)
Spec. for Structural Steel for Ships (A 131 – 55)
Spec. for Heat-Treated Carbon-Steel Bolting Material (A 261 – 54)
Spec. for Seamless and Welded Austenitic Stainless Steel Tubing for General Service (A 269 – 55)
Spec. for Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (A 271 – 55)

Standard Withdrawn:

Spec. for Carbon-Steel Castings for Valves, Flanges, and Fittings for High-Temperature Service (A 95 – 44)

Tentative Withdrawn:

Spec. for Chromium-Molybdenum Steel Plates for Boilers and Other Pressure Vessels (A 301 – 54a T)

Committee A-5 on Corrosion of Iron and Steel:

Report presented by A. P. Jahn, chairman, and the following action taken:

Accepted as Tentative:

Spec. for Zinc-Coated (Galvanized) Steel Poultry Netting (Hexagonal and Straight Line and Woven Steel Poultry Fencing (A 390 – 56 T).

Committee B-7 on Light Metals and Alloys, Cast and Wrought:

Report presented by I. V. Williams, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Aluminum-Alloy Drawn Seamless Coiled Tubes for Special Purpose Applications (B 307 – 56 T), changing Section 8 to read, "Tension test specimens shall be of the full section of the tube."

Spec. for Aluminum-Alloy Standard Structural Shapes, Rolled or Extruded (B 308 – 56 T), with the addition to the end of Section 1 on Scope of the words "such as angles, zees, channels, and I-beams," and a change in Table I to indicate the iron, per cent for alloy SG11B as 0.50.

Accepted as Tentative, Revisions in:

Spec. for Aluminum-Base Alloy Sand Castings (B 26 – 55 T)

Spec. for Magnesium-Base Alloy Sand Castings (B 80 – 55 T)

Spec. for Magnesium-Base Alloy Sheet (B 90 – 55 T), changing in Table I Alloy "AZ31B" to read "AZ31A."

Spec. for Magnesium-Base Alloy Forgings (B 91 – 55 T), with a change in footnote *b* for Table II by the deletion of the words "less than 6 in. thick."

Spec. for Magnesium-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 93 – 55 T)

Spec. for Magnesium Alloy Bars, Rods, and Shapes (B 107 – 55 T)

Spec. for Aluminum-Base Alloy Permanent Mold Castings (B 108 – 55 T), with the following additional change:

Table IV.—Add Alloy SC103A with the following properties and characteristics:

Approximate Melting Range, F.	970–1080
Foundary Characteristics	
Resistance to Hot Cracking	1
Pressure Tightness	2
Fluidity	1
Solidification Shrinkage Tendency	2
Normally Heat Treated	aged only
Other Characteristics	
Resistance to Corrosion	3
Machining	3
Polishing	4
Electroplating	3
Anodizing (Appearance)	5
Chemical Oxide Coating (Protection)	3
Strength at Elevated Temperature	3
Suitability for Welding	2
Suitability for Brazing	No

Spec. for Aluminum-Alloy Sheet and Plate for Pressure Vessel Applications (B 178 – 55 T), referencing the present footnote *b* after the tensile strength values for the O temper of alloy GM40A in Table II.

Spec. for Magnesium-Base Alloy Permanent Mold Castings (B 199 – 55 T)

Spec. for Aluminum-Alloy Sheet and Plate (B 209 – 55 T), referencing the present footnote *i* after the O temper of alloy GM40A in Table II.

Spec. for Aluminum-Alloy Drawn Seamless Tubes (B 210 – 55 T)

Spec. for Aluminum-Alloy Bars, Rods, and Wire (B 211 – 55 T)

Spec. for Magnesium-Base Alloy Extruded Tubes (B 217 – 55 T)

Spec. for Aluminum-Alloy Extruded Bars, Rods, and Shapes (B 221 – 55 T)

Spec. for Aluminum-Alloy Drawn Seamless Tubes for Condensers and Heat Exchangers (B 234 – 55 T)

Spec. for Aluminum-Alloy Extruded Tubes (B 235 – 55 T)

Spec. for Aluminum Bars for Electrical Purposes (B 236 – 55 T)

Spec. for Aluminum-Alloy Pipe (B 241 – 55 T)

Spec. for Aluminum-Alloy Bars, Rods, and Shapes for Pressure Vessel Application (B 273 – 55 T)

Spec. for Aluminum-Alloy Pipe and Tube for Pressure Vessel Applications (B 274 – 55 T)

Adopted as Standard:

Method of Measuring Thickness of Anodic Coatings on Aluminum by Means of the Filmeter (B 244 – 49 T)

Rec. Practice for Temper Designation of Light Metals and Alloys, Cast and Wrought (B 296 – 54 T)

Adopted as Standard, Revisions in:

Spec. for Magnesium Ingot and Stick for Remelting (B 92 – 55)

Spec. for Aluminum-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 179 – 55)

Standard Withdrawn:

Spec. for Aluminum Ingots for Remelting (B 24 – 46)

Committee C-4 on Clay Pipe:

Report presented by J. C. Riedel, chairman, and the following action taken:

Adopted as Standard:

Spec. for Clay Flue Linings (C 315 – 53 T)

Committee C-9 on Concrete and Concrete Aggregates:

Report presented by W. H. Price, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Concrete Aggregates (C 33 – 55 T)
Test for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (C 88 – 55 T)
Spec. for Lightweight Aggregates for Insulating Concrete (C 332 – 54 T)
Test for Resistance of Concrete Specimens to Rapid Freezing and Thawing in Water (C 290 – 52 T)
Test for Resistance of Concrete Specimens to Rapid Freezing in Air and Thawing in Water (C 291 – 52 T)
Test for Resistance of Concrete Specimens to Slow Freezing and Thawing in Water or Brine (C 292 – 52 T)
Test for Resistance of Concrete Specimens to Slow Freezing in Air and Thawing in Water (C 310 – 53 T)
Test for Bleeding of Concrete (C 232 – 53 T)
Test for Compressive Strength of Molded Concrete Cylinders (C 39 – 49)
Test for Organic Impurities in Sands for Concrete (C 40 – 48)
Test for Air Content of Freshly Mixed Concrete by the Pressure Method (C 231 – 54)

Adopted as Standard:

Descriptive Nomenclature of Constituents of Natural Mineral Aggregates (C 294 – 54 T)
Spec. for Liquid Membrane-Forming Compounds for Curing Concrete (C 309 – 53 T)

Adopted as Standard, Revisions in:

Def. of Terms Relating to Concrete and Concrete Aggregates (C 125 – 48)
Spec. for Waterproof Paper for Curing Concrete (C 171 – 53)

Committee C-13 on Concrete Pipe:

Report presented by R. R. Litehiser, chairman, and the following actions taken:

Adopted as Standard, Revisions in:

Spec. for Concrete Sewer Pipe (C 14 – 55): Covering (1) adoption as standard of present tentative revisions of Sections 3, 9, 18, and 19; and (2) immediate adoption of revisions of Sections 17 (*b*), and 29 (*i*).
Spec. for Reinforced Concrete Sewer Pipe (C 75 – 55)
Spec. for Reinforced Concrete Culvert Pipe (C 76 – 55)
Spec. for Concrete Irrigation and Drainage Pipe (C 118 – 55)

Committee C-15 on Manufactured Masonry Units:

Report presented in the absence of the chairman by J. W. Caum, and the following action taken:

Adopted as Standard, Revisions in:

Methods of Sampling and Testing Concrete Masonry Units (C 140 – 55)

Committee C-18 on Natural Building Stones:

Report presented in the absence of the chairman by J. W. Caum, and the following action taken:

Accepted as Tentative, Revisions in:

Test for Durability of Slate for Roofing (C 217 – 48 T)

Committee C-20 on Acoustical Materials:

Report presented in the absence of the chairman by J. W. Caum, and the following action taken:

Accepted as Tentative:

Test for Impedance and Absorption of Acoustical Materials by the Tube Method (C 384 – 56 T)

Committee E-11 on Quality Control of Materials:

Report presented in the absence of the chairman by J. W. Caum, and accepted as a report of progress.

TWELFTH SESSION—GILLETT MEMORIAL LECTURE[5a]

TUESDAY, JUNE 19, 5:00 P.M.

SESSION CHAIRMAN: PRESIDENT C. H. FELLOWS

Fifth Gillett Memorial Lecture:

President Fellows called upon Mr. Bruce Gonser of Battelle Memorial Inst., who remarked upon the establishment of the H. W. Gillett Memorial Lecture. This lecture is sponsored jointly by the ASTM and the Battelle Memorial Inst. to perpetuate the memory of Horace W. Gillett, one of America's leading technologists, the first Director of Battelle Memorial Inst., and for many years a very active worker in the Society, through the presentation of a lecture on a subject pertaining to the development, testing, evaluation, and application of metals.

Mr. Bruce Gonser introduced D. K. Crampton, Director of Research and Development, Chase Brass and Copper Co., who presented the fifth H. W. Gillett Memorial Lecture on the subject, "Structural Chemistry and Metallurgy of Copper." The lecturer explored three aspects of microstructure of copper. The first portion covered some new work on recrystallization and grain growth of copper alloys, investigating time intervals much shorter than have been used heretofore. In the second portion the lecturer dealt with the structural chemistry of copper and copper alloys and more particularly the relations of grains and grain boundaries both in pure copper and some copper alloys. Lastly Mr. Crampton described new investigations of the fundamentals of corrosion and corrosion resistance of copper and some of the factors affecting the incidence and growth of corrosion pits.

President Fellows expressed appreciation to Mr. Crampton for his outstanding lecture and presented to him on behalf of the Society the H. W. Gillett Memorial Lecture Certificate.

THIRTEENTH SESSION—METALS

(Continued from Second Session)

TUESDAY, JUNE 19, 8:00 P.M.

SESSION CHAIRMAN: P. V. GARIN

Correlation of Torsional and Tensile Prestrain Effects on Fracture Properties—I. Rozalsky, presented by the author.

Factors Affecting the Forming Properties of Several Copper Alloys in Strip Form—John T. Richards and Ellsworth M. Smith, presented by Mr. Richards.

The Effect of Irradiation on the Mechanical Properties of Zirconium—R. S. Kemper, Jr. and W. S. Kelly, presented by Mr. Kemper.

The Effect of Brittle Skins on the Ductility of Metals—G. W. Form and W. M. Baldwin, Jr., presented by Mr. Form.

Notch Tensile Behavior of Face-Centered Cubic Metals—E. J. Ripling, presented by G. W. Form.

Materials for Helical Compression Springs for Use at Constant Deflection from 600 to 1400 F—R. G. Matters and R. E. Lochen, presented by Mr. Matters.

(Continued in the Sixteenth Session)

[5a] Issued as separate publication.

FOURTEENTH SESSION—SYMPOSIUM ON SOLDER[4]

(Continued from Fifth and Ninth Sessions)

TUESDAY, JUNE 19, 8:00 P.M.

SESSION CHAIRMAN: L. L. WYMAN

Ultrasonic Soldering of Aluminum—J. B. Jones and J. G. Thomas, presented by Mr. Thomas.

Numerical Evaluation System for Soft Solders, Fluxes, and Solderability—L. Pessel, presented by the author.

Dip-Soldered Printed Circuit Joint Characteristics—A. A. Johns, presented by the author.

The Use of Rosin and Activated Rosin Fluxes—F. C. Disque, Jr., presented by the author.

(Continued in the Fifteenth Session)

FIFTEENTH SESSION—SYMPOSIUM ON SOLDER[4]

(Symposium Continued from Fifth, Ninth, and Fourteenth Sessions)

WEDNESDAY, JUNE 20, 9:30 A.M.

SESSION CHAIRMAN: R. M. MACINTOSH

Soldering in Semiconductor Devices—W. F. Lootens, presented by the author.

Industrial Survey of Paste Solder Alloys—H. R. Williams, presented by the author.

Method for Testing and Evaluating the Joint Properties of a Copper Liner Soldered in an Aluminum Casting—M. V. Davis, presented by the author.

Non-Corrosive and Intermediate Fluxes—H. C. Sohl, presented by the author.

Symposium Summation by G. H. Harnden.

SIXTEENTH SESSION—METALS

(Continued from Second and Thirteenth Sessions)

WEDNESDAY, JUNE 20, 9:30 A.M.

SESSION CHAIRMAN: F. L. LAQUE

The Strength of Certain Stainless and Carbon Steels at Low Temperatures—S. C. Collins, F. D. Ezekiel, O. W. Sepp, and J. W. Rizika, presented by Mr. Collins.

Effect of Composition and Heat Treatment upon the Impact Properties of AISI Types 403 and 410 Stainless Steel—E. J. Whittenberger, E. R. Rosenow, and D. J. Carney, presented by Mr. Whittenberger.

Effect of Creep Stress History at High Temperatures on the Creep of Aluminum Alloys—O. D. Sherby, T. A. Trozera, and J. E. Dorn, presented by Mr. Paul Shahinian.

Notch and Smooth Bar Stress Rupture Characteristics of Several High-Strength, Heat-Resisting Alloys in the Temperature Range Between 600 and 1000 F—J. G. Sessler and W. F. Brown, Jr., presented by Mr. Brown.

Autographic Bearing-Strength Test Method and Typical Test Values on Some Magnesium Alloys at Room and Elevated Temperatures—James A. Gusack and A. A. Moore, presented by Mr. Moore.

SEVENTEENTH SESSION—COMMITTEE REPORT SESSION

Wednesday, June 20, 11:30 a.m.

Session Chairman: K. B. Woods

Committee D-20 on Plastics:

Report presented by R. K. Witt, chairman, and the following actions taken:

Accepted as Tentative:

Spec. and Methods of Test for Polychlorotrifluoroethylene Molding and Extrusion Materials (D 1430 - 56 T)
Spec. for Styrene-Acrylonitrile Copolymer Molding and Extrusion Materials (D 1431 - 6 T)
Spec. for Nonrigid Polyvinyl Chloride and Copolymer Molding and Extrusion Compounds (D 1432 - 56 T)
Test for Flammability of Flexible Thin Plastic Sheeting (D 1433 - 56 T)
Test for Gas Transmission Rate of Plastic Sheeting (D 1434 - 56 T)
Rec. Practice for Outdoor Weathering of Plastics (D 1435 - 56 T)

Accepted as Tentative, Revisions in:

Spec. for Polystyrene Molding Compounds (D 703 - 49 T)
Spec. for Melamine-Formaldehyde Molding Compounds (D 704 - 55 T)
Spec. for Methacrylate Molding and Extrusion Compounds (D 788 - 48 T)
Test for Resistance of Plastics to Chemical Reagents (D 543 - 52 T)
Nomenclature of Descriptive Terms Pertaining to Plastics (D 675 - 55 T)
Def. of Terms Relating to Plastics (D 883 - 55 T), with the withdrawal of the definition for "Compression Molding," and changes in in the definitions for Gel Point, and Syneresis to read as follows:

> *Gel Point.*—The stage at which a liquid system becomes a gel. (See second definition of Gel.)
>
> Note:—This may be caused by such factors as continuing polymerization, change in temperature, or loss of solvent.
>
> *Syneresis.*—The contraction of a gel.
>
> Note:—This is usually evidenced by the separation of a liquid. (See first definition of Gel.)

Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (D 618 - 54)
Spec. for Cast Methacrylate Plastic Sheets, Rods, Tubes and Shapes (D 702 - 46)
Test for Flammability of Plastics 0.050 in. and Under in Thickness (D 568 - 43)
Test for Flammability of Plastics 0.050 in. and Over in Thickness (D 635 - 44)

Adopted as Standard:

Spec. for Enclosures and Servicing Units for Tests Above and Below Room Temperature (D 1197 - 54 T)
Test for Impact Resistance of Plastics and Electrical insulating Materials (D 256 - 54 T)
Rec. Practice for Long-Time Creep or Stress-Relaxation of Plastics Under Tension or Compression Loads at Different Temperatures (D 674 - 51 T)
Test for Heat Distortion Temperature of Plastics (D 648 - 45 T)
Test for Resistance of Transparent Plastics to Surface Abrasion (D 1044 - 54 T)
Test for Warpage of Sheet Plastics (D 1181 - 51 T)
Test for Resistance to Abrasion of Plastic Materials (D 1242 - 52 T)

[The recommendation for the adoption as standard of the Tentative Methods of Testing Vulcanized Fibre Used for Electrical Insulation (D 619 - 54 T) was necessarily contingent upon the corresponding recommendation on the part of Committee D-9 on Electrical Insulating Materials since the methods are under the joint jurisdiction of the two committees. Committee D-9 withdrew its concurrence in the recommendation, and Committee D-20's recommendation is accordingly being withheld.]

Adopted as Standard, Revisions in:

Test for Resistance of Plastics to Accelerated Service Conditions (D 756 - 50)

Tentatives Withdrawn:

Spec. for Nonrigid Vinyl Chloride-Acetate Resin Plastics (D 742 - 46 T)
Spec. for Nonrigid Vinyl Chloride Plastics (D 744 - 49 T)

Committee C-14 on Glass and Glass Products:

Report presented in the absence of the chairman by R. E. Hess, and the following actions taken:

Adopted as Standard:

Methods of Sampling Glass Containers (C 224 - 53 T)
Methods of Testing Structural Non-Load-Bearing Cellular Glass Blocks (C 240 - 54 T)

Adopted as Standard, Revisions in:

Def. of Terms Relating to Glass and Glass Products (C 162 - 52)

Committee C-16 on Thermal Insulating Materials:

Report presented in the absence of the chairman by W. L. Gantz, secretary, and the following actions taken:

Accepted as Tentative:

Spec. for Cellular Glass Thermal Insulation for Pipes (C 381 - 56 T), with a revision in Section 5 (a) consisting of changing 13-in. to read 18-in.
Spec. for Mineral Wool Felt Insulation (Industrial Type) for Elevated Temperatures (C 382 - 56 T)
Test for Wet Adhesion of Thermal Insulating Cements to Metal (C 383 - 56 T)

Adopted as Standard:

Spec. for Cellular Asbestos Paper Thermal Insulation for Pipes (C 298 - 52 T)
Spec. for Laminated Asbestos Thermal Insulation for Pipes (C 299 - 52 T)
Spec. for Cellular Glass Insulating Block (C 343 - 54 T)
Spec. for Corkboard Thermal Insulation (C 352 - 54 T)
Test for Density of Preformed Pipe-Covering-Type Thermal Insulation (C 302 - 52 T)
Test for Density of Preformed Block-Type Thermal Insulation (C 303 - 52 T)
Test for Adhesion of Dried Thermal Insulating Cements (C 353 - 54 T)
Test for Compression Strength of Thermal Insulating Cements (C 354 - 54 T)

Adopted as Standard, Revisions in:

Def. of Terms Relating to Thermal Insulating Materials (C 168 - 51)

Tentative Withdrawn:

Test for Vibration Resistance of Preformed Thermal Insulation (C 304 - 52 T)

The committee withdrew from the report as preprinted the tentative revision of the Definitions of Terms Relating to Thermal Insulating Materials (C 168 - 51)

Committee C-21 on Ceramic Whitewares and Related Products:

Report presented in the absence of the chairman by R. E. Hess, and the following actions taken:

Accepted as Tentative

Def. of Terms Relating to Ceramic Whitewares and Related Products (C 242 - 56 T)

Adopted as Standard:

Methods of Sampling Ceramic Whiteware Clays (C 322 - 53 T)
Method of Chemical Analysis of Ceramic Whiteware Clays (C 323 - 53 T)
Test for Free Moisture in Ceramic Whiteware Clays (C 324 - 53 T)
Test for Wet Sieve Analysis of Ceramic Whiteware Clays (C 325 - 53 T)
Method for Determination of Drying and Firing Shrinkages of Ceramic Whiteware Clays (C 326 - 53 T)
Test for Linear Thermal Expansion of Fired Ceramic Whiteware Materials by the Interferometric Method (C 327 - 53 T)
Test for Flexural Properties of Fired Dry-Pressed Whiteware Specimens at Normal Temperatures (C 328 - 53 T)
Test for Specific Gravity of Fired Ceramic Whiteware Materials (C 329 - 53 T)
Test for Impact Resistance of Ceramic Tableware (C 368 - 55 T)
Test for Modulus of Rupture of Fired, Cast or Extruded Whiteware Products (C 369 - 55 T)
Test for Moisture Expansion of Fired Whiteware Products (C 370 - 55 T)
Test for Sieve Analysis of Nonplastic Pulverized Ceramic Materials (C 371 - 55 T)
Test for Linear Thermal Expansion of Fired Whiteware Products by the Dilatometer Method (C 372 - 55 T)
Test for Water Absorption, Bulk Density, Apparent Porosity, and Apparent Specific Gravity of Fired Porous Whiteware Products (C 373 - 55 T)

Adopted as Standard, Revisions in:

Def. of Terms Relating to Ceramic Whiteware and Similar Products (C 242 - 55)

Committee C-22 on Porcelain Enamel:

Report presented in the absence of the chairman by R. E. Hess, and the following actions taken:

Accepted as Tentative:

Test for Thermal Shock Resistance of Porcelain-Enameled Utensils (C 385 - 56 T)

Accepted as Tentative, Revisions in:

Def. of Terms Relating to Porcelain Enamel (C 286 - 55 T)

Paper:

A Method for Evaluating Surface Flammability of Materials Using a Radiant Energy Source—A. F. Robertson, D. Gross, and J. Loftus, presented by Mr. Gross.

EIGHTEENTH SESSION—SYMPOSIUM ON TENSION TESTING OF NON-METALLIC MATERIALS

WEDNESDAY, JUNE 20, 2:00 P.M.

SESSION CHAIRMAN: A. C. WEBBER

Committee E-1 on Methods of Testing:

Report presented by J. R. Townsend, chairman, and the following action taken:

Adopted as Standard, Revisions in:

Spec. for ASTM Thermometers (E 1 - 55).

Symposium on Tension Testing of Non-Metallic Materials[6]

Introduction, by A. C. Webber, Symposium Chairman.

Present Practices in Tensile-Strength Testing of Paper in Industry—John Fachet and F. T. P. Plimpton, Jr., presented by Mr. Fachet.

Tension Testing of Adhesives—A. G. H. Dietz, presented by the author.

Tensile Load-Deformation Testing of Textile Structures—Stanley Backer, presented by the author (not published).

Tension Testing of Rubber—H. Tangenberg, presented by the author.

Tension Testing of Plastics—Frank W. Reinhart, presented by the author.

Tension Test Methods for Wood, Wood-Base Materials and Sandwich Constructions—L. J. Markwardt and W. G. Youngquist, presented by Mr. Youngquist.

Summary, by A. C. Webber, Symposium Chairman.

NINETEENTH SESSION—COMMITTEE REPORT SESSION

WEDNESDAY, JUNE 20, 4:00 P.M.

SESSION CHAIRMAN: VICE-PRESIDENT R. T. KROPF

Committee A-7 on Malleable-Iron Castings:

Report presented by W. A. Kennedy, chairman, and accepted as a report of progress.

Committee A-9 on Ferro-Alloys:

Report presented by W. C. Bowden, chairman, and accepted as a report of progress.

The committee withdrew from the report as preprinted the recommendation for the adoption as standard of Tentative Specification for Ferromanganese (A 99 - 53 T).

Committee B-2 on Non-Ferrous Metals and Alloys:

Report presented by B. W. Gonser, chairman, and accepted as a report of progress.

Committee B-6 on Die-Cast Metals and Alloys:

Report presented by W. Babington, chairman, and the following action taken:

Accepted as Tentative, Revisions in:

Spec. for Aluminum-Base Alloy Die Castings (B 85 - 54 T)

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[6] Issued as separate publication *ASTM STP No. 194.*

The committee withdrew from the report as preprinted the revisions of the Tentative Specification for Zinc-Base Alloy Die Castings (B 86 - 53 T).

Committee D-1 on Paint, Varnish, Lacquer and Related Products:

Report presented by W. T. Pearce, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Safflower Oil (D 1392 - 56 T)
Test for Flash Point of Drying Oils (D 1393 - 56 T)
Methods for Chemical Analysis of White Titanium Dioxide Pigments (D 1394 - 56 T)
Test for Abrasion Resistance of Clear Floor Coatings (D 1395 - 56 T)
Methods for Analysis of Polyvinyl Butryal (D 1396 - 56 T)
Test for Unsaponifiable Matter in Alkyd Resins and Resin Solutions (D 1397 - 56 T)
Test for Fatty Acid Content of Alkyd Resins and Resin Solutions (D 1398 - 56 T)
Methods for Measurement of Dry Film Thickness of Nonmetallic Coatings of Paint, Varnish, Lacquer, and Related Products Applied on a Nonmagnetic Metal Base (D 1400 - 56 T)
Test for Unsaponifiable Content of Tricresyl Phosphate (D 1399 - 56 T)

Accepted as Tentative, Revisions in:

Methods of Chemical Analysis of White Pigments (D 34 - 51 T)
Test for Distillation Range of Lacquer Solvents and Diluents (D 1078 - 49 T)
Test for Nonvolatile Content of Resin Solutions (D 1259 - 53 T)
Test for Flash Point of Volatile Flammable Materials by Tag Open-Cup Apparatus (D 1310 - 55 T)
Def. of Terms Relating to Paint, Varnish, Lacquer, and Related Products (D 16 - 52)
Methods of Testing Varnishes (D 154 - 53), with the heading preceding Section 30 changed from "Acid Number" to read "Acid Value," also the word "approximate" added before 0.1 *N* Potassium Hydroxide.
Methods of Testing Drying Oils (D 555 - 54)
Spec. for Raw Soybean Oil (D 124 - 48)

Adopted as Standard:

Test for No-Dirt-Retention Time of Traffic Paint (D 1297 - 53 T)
Test for Phthalic Anhydride Content of Alkyd Resins and Esters Containing Other Dibasic Acids (D 1306 - 54 T)
Test for Phthalic Anhydride Content of Alkyd Resins and Esters Containing Other Dibasic Acids (Spectrophotometric) (D 1307 - 54 T)
Test for Settling Properties of Traffic Paints During Storage (D 1309 - 54 T)
Test for Apparent Free Phenols in Synthetic Phenolic Resins or Solutions Used for Coating Purposes (D 1312 - 54 T)
Test for Nonvolatile Matter in Lacquer Solvents and Diluents (D 1353 - 54 T)
Methods of Testing Varnishes (D 154 - 53 T), to be added to present Standard D 154 - 53
Method for Evaluating Degree of Blistering of Paints (D 714 - 54 T)
Rec. Practice for Conducting Exterior Exposure Tests of Paints on Wood (D 1006 - 51 T)
Spec. for Asphalt-Base Emulsions for Use as Protective Coatings for Metal (D 1187 - 51 T)

Adopted as Standard, Revisions in:

Test for Flash Point by Tag Closed Tester (D 56 - 52), jointly with Committee D-2.
Spec. for Ethylene Glycol Monobutyl Ether (D 330 - 35)
Spec. for Ethylene Glycol Monoethyl Ether (D 331 - 35)
Spec. for Acetate Ester of Ethylene Glycol Monoethyl Ether (95 to 96 per cent Grade) (D 343 - 35)
Spec. for Tricresyl Phosphate (D 363 - 49)
Spec. for Methyl Ethyl Ketone (D 740 - 46)

Accepted for Publication as Information Only:

Methods for Preparation of Aluminum and Aluminum Alloy Surfaces for Painting
Methods for Preparation of Magnesium Alloy Surfaces for Painting

The committee withdrew from the report as preprinted the recommendation for the revision and reversion to tentative of the Standard Specifications for Copper Phthalocyanine Blue (D 963 - 49).

Committee D-4 on Road and Paving Materials:

Report presented in the absence of the chairman by A. B. Cornthwaite, and the following actions taken:

Accepted as Tentative:

Test for Water-Soluble Chlorides Present as Admixes in Graded Aggregate Road Mixes (D 1411 - 56 T) (jointly with Committee D-18)

Accepted as Tentative, Revisions in:

Spec. for Preformed Expansion Joint Fillers for Concrete (Nonextruding and Resilient Types) (D 544 - 52 T)

Spec. for Preformed Expansion Joint Fillers for Concrete, (Nonextruding and Resilient Types) (D 544 - 49)
Spec. for Emulsified Asphalt (D 977 - 53)
Spec. for Bituminous Mixing Plant Requirements (D 995 - 55)
Methods of Testing Preformed Expansion Joint Fillers for Concrete, (Nonextruding and Resilient Types) (D 545 - 49)

Adopted as Standard, Revisions in:

Test for Specific Gravity of Compressed Bituminous Mixtures (D 1188 - 53)
Test for Distillation of Tars and Tar Products (D 20 - 55)

Committee D-13 on Textile Materials:

Report presented by W. D. Appel, chairman, and the following actions taken (subject to confirming letter ballot of Committee D-13):

Accepted as Tentative:

Test for Twist in Single Spun Yarns (Untwist-Twist Method) (D 1422 - 56 T)
Test for Twist in Single Spun Yarns (Direct-Counting Method) (D 1423 - 56 T)
Test for Tear Resistance of Woven Fabrics by Falling-Pendulum Method (D 1424 - 56 T)
Test for Unevenness of Textile Strands (D 1425 - 56 T)
Test for Non-Lint Content of Cotton (Shirley Analyser Method) (D 1451 - 56 T)

Accepted as Tentative, Revisions in:

Methods of Quantitative Analysis of Textiles (D 629 - 55 T)
Test for Tensile Strength of Wool Fiber Bundles (D 1294 - 53 T)
Methods of Testing and Tolerances for Rayon Tire Cord (D 885 - 54 T)
Methods of Testing and Tolerances for Yarns Containing Wool (D 1285 - 53 T), with a change in Note 2 under Section 10(*b*) to read as follows:

> NOTE 2.—The tolerances here set forth are recommended for use in the arbitration of disputes. These tolerances apply to material in the form of spun yarn only. They are not applicable to yarn in fabric or in garments or in any state in which its form has been changed from the spun yarn.

Methods of Testing Spun End Filament Yarns Made Wholly or in Part of Man-Made Organic Base Fibers (D 1380 - 55 T)
Def. of Terms Relating to Textile Materials (D 123 - 55)

[The fiber classification chart, which had been recommended for addition to part B of the Standard Definitions D 123 - 55 has been withdrawn.

Methods of Identification of Fibers in Textiles (D 276 - 49)

Adopted as Standard:

Test for Maturity of Cotton Fibers (Random Sample-Sodium Hydroxide Swelling Method) (D 1443 - 53 T)
Test for Cross-Sectional Characteristics of Cotton Fibers (D 1444 - 53 T)
Test for Micronaire Fineness of Cotton Fibers (D 1448 - 54 T)
Spec. and Methods of Test for Fineness of Wool Tops (D 472 - 55 T)
Methods of Core Sampling of Raw Wool in Packages for Determination of Percentage of Clean Wool Fiber Present (D 1060 - 55 T)

Adopted as Standard, Revisions in:

Methods of Testing and Tolerances for Cotton Sewing Threads (D 204 - 42)
Test for Yarn Number of Yarn from Fabrics (D 1059 - 52)

Accepted for Publication as Information Only:

Test for Strength of Cotton Fibers Using a Finite Gage Length (Flat Bundle Method)

[The report had listed in error for adoption as standard the Tentative Method of Test for Wool Content of Raw Wool (D 1334 - 54 T). This method of test is being continued as tentative as stated elsewhere in the report.]

Committee D-16 on Industrial Aromatic Hydrocarbons and Related Materials:

Report presented by D. F. Gould, chairman, and the following action taken:

Adopted as Standard, Revisions in:

Test for Distillation of Industrial Aromatic Hydrocarbons (D 850 - 55)

Committee E-3 on Chemical Analysis of Metals:

Report presented in the absence of the chairman by H. A. Bright, Honorary Chairman, and the following actions taken (subject to confirming letter ballot of Committee E-3):

Accepted as Tentative:

Methods for Chemical Analysis of Titanium and Titanium-Base Alloys (E 120 - 56 T)
Methods for Chemical Analysis of Nickel-

Chromium and Nickel-Chromium-Iron Alloys (E 38 – 56 T)
Methods for Chemical Analysis of Copper-Tellurium Alloys (E 121 – 56 T)
Methods for Chemical Analysis of Chromium-Copper Alloys (E 118 – 56 T)
Methods for Chemical Analysis of Slab Zinc (Spelter) (E 40 – 56 T)
Photometric Method for Determination of Phosphorus in High-Phosphorus Brazing Alloys (E 56 – 56 T)

Accepted as Tentative, Revisions in:

Photometric Methods for Determination of Manganese in Steel (E 30 – 54 T)
Photometric Methods for Chemical Analysis of Aluminum and Aluminum-Base Alloys (E 34 – 54 T)
Photometric Methods for Chemical Analysis of Lead, Tin, Antimony, and Their Alloys (E 87 – 50 T)
Photometric Methods for Chemical Analysis of Electronic Nickel (E 107 – 54 T)
Methods for Chemical Analysis of White Metal Bearing Alloys (E 57 – 50 T)
Methods for Chemical Analysis of Zinc-Base Die-Casting Alloys (E 47 – 45)

Adopted as Standard:

Photometric Methods for Chemical Analysis of Aluminum and Aluminum-Base Alloys (E 34 – 54 T) (Sections 6 to 56 only)
Photometric Methods for Chemical Analysis of Magnesium and Magnesium-Base Alloys (E 35 – 50 T)
Photometric Method for Determination of Cobalt in Nickel (E 39 – 49 T)
Photometric Methods for Chemical Analysis of Copper and Copper-Base Alloys (E 62 – 50 T)
Methods for Chemical Analysis of Pig Lead (E 37 – 50 T)
Methods for Chemical Analysis of Lead- and Tin-Base Solder Metal (E 46 – 50 T)
Methods for Chemical Analysis of Copper-Nickel and Copper-Nickel-Zinc Alloys (E 75 – 50 T)
Methods for Chemical Analysis of Nickel-Copper Alloys (E 76 – 50 T)
Method for Polarographic Determination of Lead and Cadmium in Zinc (E 68 – 46 T)

Adopted as Standard, Revisions in:

Methods for Chemical Analysis of Steel, Cast Iron, Open-Hearth Iron, and Wrought Iron (E 30 – 52)
Methods for Chemical Analysis of Nickel-Chromium and Nickel-Chromium-Iron Alloys (E 38 – 48)

Tentative Withdrawn:

Photometric Method for Determination of Iron in Slab Zinc (Spelter) (E 64 – 50 T)

Reapproval of Standard:

Methods for Chemical Analysis of Slab Zinc (Spelter) (E 40 – 45)

Committee E-4 on Metallography:

Report presented by L. L. Wyman, chairman, and accepted as a report of progress.

Committee E-12 on Appearance:

Report presented in the absence of the chairman by P. J. Smith, and accepted as a report of progress.

TWENTIETH SESSION—MARBURG LECTURE[5a]

WEDNESDAY, JUNE 20, 4:30 P.M.

SESSION CHAIRMAN: PRESIDENT C. H. FELLOWS

Edgar Marburg Lecture:

President Fellows opened the session by giving a brief statement of the purpose of the lecture. Charles E. Reed, General Manager, Silicone Products Dept., General Electric Co., Waterford, N. Y., was then introduced by the Executive Secretary R. J. Painter as the thirtieth Edgar Marburg Lecturer. Speaking on the subject, "The Industrial Chemistry, Properties, and Applications of Silicones," Mr. Reed discussed the basic chemical structure of silicones with particular reference to their similarities and differences compared with other industrially important polymeric substances, and the physical and chemical properties of silicones leading to their industrially important applications. He then took up processes of manufacture, the important types of silicones and their applications, ending with a look into the future of silicones in selected industries such as electrical, aircraft, protective coating, and textile.

President Fellows expressed appreciation to Mr. Reed for his excellent lecture and presented to him on behalf of the Society the Edgar Marburg Lecture Certificate.

TWENTY-FIRST SESSION—FATIGUE

THURSDAY, JUNE 21, 9:30 A.M.

SESSION CHAIRMAN: R. E. PETERSON

Report of Committee E-9 on Fatigue:

Report presented by R. E. Peterson, chairman, and accepted as a report of progress.

Fatigue Properties of Comparable Cast and Wrought Steel—C. W. Briggs, L. J. Ebert, and E. B. Evans, presented by Mr. Ebert.

Effect of Changing Cyclic Modulus on the Bending Fatigue Strength—A. A. Blatherwick and B. J. Lazan, presented by Mr. Blatherwick.

Fatigue Crack Propagation in Aluminum Alloys—M. S. Hunter and W. G. Fricke, Jr., presented by Mr. Fricke.

The Notched Fatigue Properties of Some Titanium Alloys—M. J. Sinnott, L. Thomassen, and A. W. Demmler, presented by Mr. Demmler.

(*Continued in the Twenty-Third Session*)

TWENTY-SECOND SESSION—COMMITTEE REPORT SESSION

THURSDAY, JUNE 21, 11:30 A.M.

SESSION CHAIRMAN: L. J. JACOBI

Committee C-12 on Mortars for Unit Masonry:

Report presented by R. E. Copeland, chairman, and accepted as a report of progress.

Committee D-7 on Wood:

Report presented in the absence of the chairman by W. G. Youngquist, and the following actions taken:

Accepted as Tentative:

Method of Testing Wood Preservatives by Laboratory Soil-Block Cultures (D 1413 – 56 T)

Accepted as Tentative, Revisions in:

Test for Evaluating the Properties of Building Fiberboards (D 1037 – 52 T)

Adopted as Standard:

Spec. for Copperized Chromated Zinc Chloride (D 1271 – 53 T)

Spec. for Pentachlorophenol (D 1272 – 53 T)

Spec. for Ammoniacal Copper Arsenite (D 1325 – 54 T)

Method for Chemical Analysis of Copperized Chromated Zinc Chloride (D 1273 – 53 T)

Method for Chemical Analysis of Pentachlorophenol (D 1274 – 53 T)

Method for Chemical Analysis of Ammoniacal Copper Arsenite (D 1326 – 54 T)

Test for Ash in Wood (D 1102 – 50 T)

Test for Holocellulose in Wood (D 1104 – 50 T)

Test for Lignin in Wood (D 1106 – 50 T)

Test for Alcohol-Benzene Solubility of Wood (D 1107 – 50 T)

Test for Ether Solubility of Wood (D 1108 – 50 T)

Test for One Per Cent Caustic Soda Solubility of Wood (D 1109 – 50 T)

Test for Water Solubility of Wood (D 1110 – 50 T)

Method for Preparation of Extractive-free Wood (D 1105 – 50 T)

Committee D-12 on Soaps and Other Detergents:

Report presented in the absence of the chairman by M. D. Huber, and the following actions taken:

Accepted as Tentative, Revisions in:

Methods for Sampling and Analysis of Sodium Triphosphate (D 501 - 55 T)

Adopted as Standard:

Test for pH of Aqueous Solutions of Soaps and Detergents (D 1172 - 54 T)
Test for Surface and Interfacial Tension of Solutions of Surface-Active Agents (D 1331 - 54 T)

Adopted as Standard, Revisions in:

Def. of Terms Relating to Soaps and Other Detergents (D 459 - 55)

Committee D-14 on Adhesives:

Report presented in the absence of the chairman by L. C. Gilbert, and the following action taken:

Accepted as Tentative, Revisions in:

Method for Determining the Effect of Moisture and Temperature on Adhesive Bonds (D 1151 - 51 T)

Adopted as Standard:

Test for Storage Life of Adhesives by Consistency and Bond Strength (D 1337 - 54 T)
Test for Working Life of Liquid or Paste Adhesives by Consistency and Bond Strength (D 1338 - 54 T)

Committee D-21 on Wax Polishes and Related Materials:

Report presented in the absence of the chairman by L. C. Gilbert, and the following actions taken:

Accepted as Tentative

Test for Flash Point of Solvent-Type Liquid Waxes (D 1437 - 56 T)
Methods for Application of Emulsion Floor Polishes to Substrates for Testing Purposes (D 1436 - 56 T)

Accepted as Tentative, Revisions in:

Test for Paraffin-Type Hydrocarbons in Carnauba Wax (D 1342 - 54 T)

Committee D-22 on Atmospheric Sampling and Analysis:

Report presented in the absence of the chairman by F. Y. Speight, and accepted as a report of progress.

Committee D-23 on Cellulose and Cellulose Derivatives:

Report presented in the absence of the chairman by L. C. Gilbert, and the following actions taken:

Accepted as Tentative:

Test for Pentosans in Cellulose (Aniline Acetate Colorimetric Method) (D 1438 -56 T)
Methods of Testing Sodium Carboxymethylcellulose (D 1439 - 56 T)

Adopted as Standard:

Spec. and Methods of Test for Soluble Cellulose Nitrate (D 301 - 54 T)
Test for Viscosity of Cellulose Derivatives by Ball-Drop Method (D 1343 - 54 T)
Method of Testing Cellulose Acetate (D 871 - 54 T)
Method of Testing Methylcellulose (D 1347 - 54 T)
Test for Moisture in Cellulose (D 1348 - 54 T)

Committee E-10 on Radioactive Isotopes:

Report presented in the absence of the chairman by L. C. Gilbert, and accepted as a report of progress.

TWENTY-THIRD SESSION—FATIGUE

(Continued from Twenty-First Session)

THURSDAY, JUNE 21, 2:00 P.M.

SESSION CHAIRMAN: F. M. HOWELL

Strengths and Failure Characteristics of AMA 5534A (S-816) Alloy in Direct Tensile Fatigue at Elevated Temperatures—Charles A. Hoffman, presented by the author.

Statistical Analysis of Fatigue Data—R. Roeloffs and F. Garofalo, presented by Mr. Richard W. Whitmore.

An Evaluation of Statistical Treatments of Fatigue Data—M. N. Torrey and G. R. Gohn, presented by Miss Torrey.

A Wire-Fatigue Machine for Investigation of the Influence of Complex Stress Histories—H. T. Corten and G. M. Sinclair, presented by Mr. Corten.

TWENTY-FOURTH SESSION—HEAT-RESISTANT AND CORROSION-RESISTANT ALLOYS

THURSDAY, JUNE 21, 2:00 P.M.

SESSION CHAIRMAN: PAST-PRESIDENT H. L. MAXWELL

Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel, and Related Alloys:

Report presented by Jerome Strauss, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Seamless and Welded Austenitic Stainless Steel Pipe (A 312 – 55 T)

[The revision of Specifications A 312 – 55 T requires joint action of Committees A-1 and A-10. Since Committee A-1 has withdrawn its recommendation that grades TP304L and TP316L be added to the specifications, these grades will not be added at this time.]

Adopted as Standard, Revisions in:

Spec. for Seamless and Welded Austenitic Stainless Steel Tubing for General Service (A 269 – 55)

Spec. for Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (A 271 – 55)

Advisory Committee on Corrosion:

Report presented by Jerome Strauss, chairman, and accepted as a report of progress.

Joint Committee on Effect of Temperature on the Properties of Metals:

Report presented by F. B. Foley, chairman, and accepted as a report of progress.

Papers:

The Corrosion Properties of Chromium-Nickel-Manganese Austenitic Stainless Steels—W. G. Renshaw and R. A. Lula, presented by Mr. Renshaw.

Fretting Wear of Zircaloy-2 Pellets in High-Temperature Water—L. A. Waldman and P. Cohen, presented by Mr. Waldman.

Intergranular Corrosion Resistance of Low-Carbon Austenitic Chromium-Manganese-Nickel Steels—W. O. Binder, J. T. Thompson, and C. R. Bishop, presented by Mr. Binder.

The Mechanical Properties of Type 201 Chromium-Nickel Manganese Stainless Steel Sheet—R. A. Walsh, R. L. Cook, and R. A. Lula, presented by Mr. Walsh.

The new AISI Types 201 (17-4-6) and 202 (18-5-8) Production and Fabrication—G. W. Hinkle, presented by the author.[7]

Influence of Hot-Working Conditions on the High-Temperature Strength Properties of a Heat-Resistant Alloy—J. F. Ewing and J. W. Freeman, presented by Mr. Ewing

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[7] Published in the ASTM BULLETIN, No. 220, February 1957, p. 47 (TP 35).

TWENTY-FIFTH SESSION—COMMITTEE REPORT SESSION

THURSDAY, JUNE 21, 4:30 P.M.

SESSION CHAIRMAN: E. J. ALBERT

Committee B-1 on Wires for Electrical Conductors:

Report presented in the absence of the chairman by A. A. Jones, secretary, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Tinned Soft or Annealed Copper Wire for Electrical Purposes (B 33 - 53 T)
Spec. for Lead-Coated and Lead-Alloy-Coated Soft Copper Wire for Electrical Purposes (B 189 - 53 T)
Spec. for Concentric-Lay-Stranded Aluminum Conductors, Steel-Reinforced (B 232 - 55 T)
Spec. for Tinned Hard-Drawn and Medium-Bard-Drawn Copper Wire for Electrical Purposes (B 246 - 51 T)
Method for Determination of Cross-Sectional Area of Stranded Conductors (B 263 - 53 T)
Spec. for Silver-Coated Soft or Annealed Copper Wire (B 298 - 55 T)

Adopted as Standard:

Spec. for Hard-Drawn Copper Wire (B 1 - 53 T)
Spec. for Soft or Annealed Copper Wire (B 3 - 54 T)

Adopted as Standard, Revisions in:

Spec. for Concentric-Lay-Stranded Copper Conductors, Hard, Medium-Hard, or Soft (B 8 - 53)
Spec. for Cored, Annular, Concentric-Lay-Stranded Copper Conductors (B 226 - 52)
Spec. for Concentric-Lay-Stranded Copper Covered Steel Conductors (B 228 - 52)
Spec. for Concentric-Lay-Stranded Copper and Copper Covered Steel Composite Conductors (B 229 - 52)
Spec. for Concentric-Lay-Stranded Aluminum Conductors, Hard-Drawn and Three-Quarter Hard-Drawn (B 231 - 55)
Spec. for Three-Quarter Hard Aluminum Wire for Electrical Purposes (B 262 - 55)

Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys:

Report presented in the absence of the chairman by A. W. Tracy, secretary, and accepted as a report of progress.

Committee B-4 on Metallic Materials for Electrical Heating, Electrical Resistance and Electrical Contacts:

Report presented in the absence of the chairman by R. E. Hess, and the following actions taken:

Adopted as Standard:

Test for Maximum Loading Stress at Temperature of Thermostat Metals (Cantilever Beam Method) (B 305 - 56)
Test for Flexivity of Thermostat Metals (B 106 - 51 T)
Test for Modulus of Elasticity of Thermostat Metals (Cantilever Beam Method) (B 223 - 51 T)

Adopted as Standard, Revisions in:

Test for Change of Resistance with Temperature of Metallic Materials for Electrical Heating (B 70 - 39)

Standard Withdrawn:

Test for Equivalent Yield Stress of Thermostat Metals (B 191 - 50)

Committee B-5 on Copper and Copper Alloys, Cast and Wrought:

Report presented by G. H. Harnden, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Copper Drainage Tube (DWV) (B 306 - 56 T), changing in Section 11 (*e*) the word "nominal" to read "standard."

Accepted as Tentative, Revisions in:

Spec. for General Requirements for Wrought Seamless Copper and Copper-Alloy Pipe and Tube (B 251 - 55 T)
Spec. for Threadless Copper Pipe (B 302 - 55 T)

Adopted as Standard:

Spec. for Copper and Copper-Alloy Die Forgings (Hot Pressed) (B 283 - 53 T)
Spec. for Nickel-Tin Bronze Castings (B 292 - 54 T)

Adopted as Standard, Revisions in:

Spec. for Brass Plate, Sheet, Strip, and Rolled Bar (B 36 - 55)

Spec. for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 - 54)

Spec. for Seamless Copper Bus Pipe and Tube (B 188 - 55)

Spec. for Copper-Nickel-Zinc Alloy (Nickel Silver) Wire (B 206 - 54), in Table I with the chemical requirements for copper for alloy E expressed as 63.5 to 66.5, and the chemical requirements for nickel for alloy E expressed as 9.0 to 11.0.

After presenting the report, the chairman indicated some objection had been raised to the second sentence under the activities of Subcommittee G-1 and he presented the following substitute wording: The subcommittee calls attention to the fact that it has not yet had an opportunity to determine whether the revised unified Tentative Methods for Determining the Average Grain Size of Metals (E 112) meets the needs of the copper and brass industry and has asked Committee B-5 to discuss this matter officially with Committee E-4.

On motion, duly seconded, this substitute wording was approved by the Society.

Committee B-8 on Electrodeposited Metallic Coatings:

Report presented in the absence of the chairman by R. E. Hess, and accepted as a report of progress.

Committee B-9 on Metal Powders and Metal Powder Products:

Report presented by J. L. Bonanno, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for High-Density Iron, Sintered Metal Powder Structural Parts (B 309 - 56 T)

Spec. for Low- and Medium-Density Iron, Sintered Metal Powder Structural Parts (B 310 - 56 T)

Test for Green Strength of Compacted Metal Powder Specimens (B 312 - 56 T)

Test for Density of Cemented Carbides (B 311 - 56 T)

Adopted as Standard, Revisions in:

Test for Sieve Analysis of Granular Metal Powders (B 214 - 48)

Committee C-1 on Cement:

Report presented by R. R. Litehiser, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Portland Blast-Furnace Slag Cement (C 205 - 53 T)

Test for Calcium Sulfate in Hydrated Portland Cement Mortar (C 265 - 55 T)

Test for False Set of Portland Cement (C 359 - 55 T)

Methods of Chemical Analysis of Portland Cement (C 114 - 51 T)

Adopted as Standard:

Test for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. Cube Specimens) (C 109 - 54 T)

Adopted as Standard, Revisions in:

Spec. for Portland Cement (C 150 - 55)

Spec. for Air-Entraining Portland Cement (C 175 - 55)

Test for Autoclave Expansion of Portland Cement (C 151 - 54)

Standard Withdrawn:

Test for Soundness of Hydraulic Cement Over Boiling Water (Pat Test) (C 189 - 49)

Committee C-2 on Magnesium Oxychloride and Magnesium Oxysulfate Cements:

Report presented in the absence of the chairman by R. E. Hess, and accepted as a report of progress.

Committee D-5 on Coal and Coke:

Report presented by W. W. Anderson, chairman, and the following actions taken:

Accepted as Tentative:

Test for Equilibrium Moisture of Coal at 96 to 97 Per Cent Relative Humidity and 30 C (D 1412 - 56 T)

Accepted as Tentative, Revisions in:

Def. of Terms Relating to Coal and Coke (D 121 - 30)

Committee D-17 on Naval Stores:

Report presented by V. E. Grotlisch, chairman, and the following action taken:

Adopted as Standard:

Test for Unsaponifiable Matter in Rosin (D 1065 - 54 T)

TWENTY-SIXTH SESSION—SYMPOSIUM ON STEAM QUALITY

THURSDAY, JUNE 21, 8:00 P.M.

SESSION CHAIRMAN: M. D. BAKER

Committee D-19 on Industrial Water:

Report presented by Max Hecht, chairman, and the following actions taken:

Accepted as Tentative:

Test for Ammonia in Industrial Water and Industrial Waste Water (D 1426 - 56 T)
Test for Sodium and Potassium Ions in Industrial Water and Water-formed Deposits by Flame Photometry (D 1428 - 56 T)
Test for Specific Gravity of Industrial Water and Industrial Waste Water (D 1429 - 56 T)
Test for Residual Chlorine in Industrial Waste Water (D 1427 - 56 T)

Adopted as Standard:

Spec. for Reagent Water (D 1193 - 55 T)
Test for Oily Matter in Industrial Waste Water (D 1340 - 54 T)

Adopted as Standard, Revisions in:

Def. of Terms Relating to Industrial Water and Industrial Waste Water (D 1129 - 55)

Accepted for Publication as Information Only:

Tests for Appearance Properties of Industrial Water and Industrial Waste Water

Max Hecht Award:

The third Max Hecht Award was presented to A. A. Berk, U. S. Bureau of Mines, Industrial Water Branch, College Park, Md.

This award is presented in recognition of outstanding service to Committee D-19 on Industrial Water in the advancement of its objective—the study of water as an engineering material. The award was established by the committee in 1954 in honor of its first chairman, Max Hecht.

Symposium on Steam Quality[8]

(*Sponsored by Joint Research Committee on Boiler Feedwater Studies*)

Measurement and Purification of Steam to 0.01 ppm Total Dissolved Solids—W. B. Gurney, presented by the author.
Steam Purity Determination by Tracer Techniques—E. E. Coulter and T. M. Campbell, Jr., presented by Mr. Campbell.
Comments on Corrections to Steam Conductivity Measurements—R. O. Parker and R. J. Ziobro, presented by Mr. Parker.
Construction and Operation of Larson-Lane Steam Purity and Condensate Analyzers—A. B. Sisson, F. G. Straub, and R. W. Lane, presented by Mr. Lane.

TWENTY-SEVENTH SESSION—GENERAL TESTING

THURSDAY, JUNE 21, 8:00 P.M.

SESSION CHAIRMAN: PAST-PRESIDENT J. R. TOWNSEND

Effect of Geometry on the Properties of C4A-T6 and S970A-T6 Aluminum Alloy Castings—W. H. Johnson and H. F. Bishop, presented by Mr. Johnson.
The Effect of Static Mean Stress on the Damping Properties of Materials—Neal L. Person and Benjamin J. Lazan, presented by Mr. A. A. Blatherwick.
An Ultrasonic Scanning and Recording System—W. J. MacGonnagle and W. N. Beck, presented by Mr. MacGonnagle.
Evaluation of Test Variables in the Determination of Shear Strength—R. W. Fenn, Jr. and R. B. Clapper, presented by Mr. Fenn.
Crack Initiation and Propagation in the V-Notch Charpy Impact Specimen—Carl E. Hartbower, presented by the author.

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[8] Issued as separate publication *ASTM STP No. 192.*

TWENTY-EIGHT SESSION—SOIL

THURSDAY, JUNE 21, 8:00 P.M.

SESSION CHAIRMAN: MILES N. CLAIR

From Experiments with Gamma Ray Transmission Through Soils—R. K. Bernhard, M. Chasek, and P. Griggs, presented by Mr. Bernhard.

Evaluation of Lime for Stabilization of Loess—J. G. Laguros, D. T. Davidson, R. L. Handy, and T. Y. Chu, presented by Mr. Laguros.

Field and Laboratory Determinations of the Bearing Capacity of Hard Pan for Design of Deep Foundations—W. S. Housel, presented by the author.

Applications of Environmental Testing of Soils—Donald M. Burmister, presented by the author.

Simple Charts Illustrating Relationships Between Moisture Content, Density, and Voids Properties of Soils—N. W. McLeod, presented by the author.

TWENTY-NINTH SESSION—INFORMAL PRESENTATION OF VARIOUS COMPLEX RHEOLOGICAL PROBLEMS

(*Sponsored jointly by Society of Rheology and ASTM Committee E-1*

FRIDAY, JUNE 22, 9:30 A.M.

SESSION CHAIRMAN: W. H. MARKWOOD, JR.

The following series of presentations presents recent steps taken toward the solution of different kinds of complex rheological problems. They cover broad and diverse topics involving the kinds of developmental approaches in research techniques, interpretive as well as experimental, required toward the successful solution of problems of stress, strain and flow in solids and semisolids.

The purpose of the symposium was to develop an increased awareness that rheology plays an important research role in many fields beyond a study of methods for the measurement of the viscosity of fluids.

Dynamic Viscosity and Shear Modulus of Molten Polystyrene as a Function of Molecular Weight—W. P. Cox, L. E. Nielsen, and R. Keeney, presented by Mr. Cox.

Stress-Strain Relations in Plastic and Frictional Soils—D. C. Drucker, presented by the author.

Rheology of Asphalts—W. Philippoff, presented by the author.[9]

The Impact-Absorbing Capacity of Textile Yarns—J. C. Smith, F. L. McCrackin, and H. F. Schiefer, presented by Mr. McCrackin.[10]

The Characterization of Pressure-Sensitive Adhesives—F. H. Wetzel, presented by the author.[11]

A New Coal Plastometer—D. C. Hills, presented by the author.[12]

Photographic Method for Sharpening Isochromatic Photoelastic Patterns—presented by R. R. Myers.

Stresses and Strains in The Shaping of Metal Tubes—E. T. Onat and W. Prager, presented by Mr. Prager.

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[9] To be released for publication in a technical journal.

[10] Published in the ASTM BULLETIN No. 220, February 1957, p. 52 (TP 49).

[11] Published in the ASTM BULLETIN No. 221, April 1957, p. 64 (TP 72).

[12] Published in the ASTM BULLETIN No. 218, December 1956, p. 34 (TP 212).

THIRTIETH SESSION—SYMPOSIUM ON IN-PLACE SHEAR TESTING OF FOUNDATION SOIL BY THE VANE METHOD[13]

FRIDAY, JUNE 22, 9:30 A.M.

SESSION CHAIRMAN: J. O. OSTERBERG

Introduction—Vane Testing for Shear Properties of Soil—The Present Status of Vane Testingby J. O. Osterberg, Symposium Chairman.

An Apparatus and Method of Vane Shear Testing of Soils—Harold J. Gibbs, presented by the author.

Deep Vane Tests in Gulf of Mexico—Carl W. Fenske, presented by the author.

Vane In-Place Soil Shear Measuring Device—W. A. Hill, presented by Mr. George F. Sowers.

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[13] Issued as separate publication *ASTM STP No. 193*.

THIRTY-FIRST SESSION—COMMITTEE REPORT SESSION

FRIDAY, JUNE 22, 12:00 NOON

SESSION CHAIRMAN: L. S. REID

Committee A-3 on Cast Iron:

Report presented by H. W. Stuart, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Cast Nodular Iron for Pressure-Containing Parts for Use at Elevated Temperatures (A 395 - 55 T)

Adopted as Standard, Revisions in:

Spec. for Gray Iron Castings (A 48 - 48)

Spec. for Gray Iron Castings for Pressure-Containing Parts for Temperatures Up to 650 F (A 278 - 53), changing in Section 4(*b*) the words "greater than 2 in. in thickness" to read "2 in. or more in thickness."

Committee D-6 on Paper and Paper Products:

Report presented by W. R. Willets, chairman, and the following actions taken:

Adopted as Standard:

Methods of Testing Analytical Filter Papers (D 981 - 51 T)

Committee D-9 on Electrical Insulating Materials:

Report presented in the absence of the chairman by H. K. Graves, vice-chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Methods of Conditioning Plastics and Electrical Insulating Materials (D 618 - 54)

Methods of Testing Solid Filling and Treating Compounds Used for Electrical Insulation (D 176 - 44)

Adopted as Standard:

Methods of Testing Askarels (D 901 - 55T)

Spec. for Enclosures and Servicing Units for Tests Above and Below Room Temperature (D 1197 - 54 T)

Adopted as Standard, Revisions in:

Method of Testing Laminated Tubes Used for Electrical Insulation (D 348 - 52)

Method of Testing Laminated Round Rods Used for Electrical Insulation (D 349 - 52)

Method of Sampling Electrical Insulating Oils (D 923 - 49)

Methods of Testing Varnished Glass Fabrics and Varnished Glass Fabric Tapes Used for Electrical Insulation (D 902 - 53)

Accepted for Publication as Information Only:

Spec. for Electrical Insulating Paper Sulfate (Kraft) Tissue for Capacitor Dielectric

The committee withdrew from the report as preprinted the recommendations for the adoption as standard of Tentative Method of Testing Vulcanized Fibre Used for Electrical Insulation (D 619 - 54 T), and Rec. Practice for Purchase of Uninhibited Mineral Oil for Use in Transformers and in Circuit Breakers (D 1040 - 54 T).

Committee D-10 on Shipping Containers:

Report presented in the absence of the chairman by S. F. Etris, and the following action taken:

Accepted as Tentative, Revisions in:

Def. of Terms Relating to Shipping Containers (D 996 - 50)

Committee E-2 on Emission Spectroscopy:

Report presented in the absence of the chairman by S. F. Etris, and the following actions taken:

Accepted as Tentative:

Method for Spectrochemical Analysis of Pig Lead by the Point-to-Plane Spark Technique (E 117 - 56 T)

Rec. Practice for Photographic Processing in Spectrochemical Analysis (E 115 - 56 T)

Rec. Practice for Photographic Photometry in Spectrochemical Analysis (E 116 - 56 T)

Accepted for Publication as Information Only:

Suggested Method for Spectrochemical Analysis of Aluminum and Its Alloys by the Pin-to-Pin Spark Technique

Suggested Method for Spectrochemical Analysis of Glass for Alkali Elements by Flame Photometry

Committee E-7 on Nondestructive Testing:

Report presented by J. H. Bly, chairman, and accepted as a report of progress.

Committee E-14 on Mass Spectrometry:

Report presented in the absence of the chairman by A. B. Kent, secretary, and accepted as a report of progress.

Committee F-1 on Materials for Electron Tubes and Semiconductor Devices:

Report presented in the absence of the chairman by F. Y. Speight, and the following actions taken:

Accepted as Tentative, Revisions in:

Methods of Testing Sleeves and Tubing for Radio-Tube Cathodes (F 128 – 55 T)

Spec. for Molybdenum Wire Under 20 Mils in Diameter (F 289 – 54 T)

Test for Diameter by Weighing of Fine Wire Used in Electronic Devices and Lamps (F 205 – 55 T)

Adopted as Standard:

Spec. for Round Chromium-Copper Wire for Electronic Devices (F 268 – 53 T)

Test for Sag of Tungsten Wire (F 269 – 52 T)

Test for Relative Thermionic Emissive Properties of Materials Used in Electron Tubes (F 270 – 52 T)

THIRTY-SECOND SESSION—COMMITTEE REPORT SESSION

FRIDAY, JUNE 22, 12:00 NOON

SESSION CHAIRMAN: VICE-PRESIDENT R. A. SCHATZEL

Committee C-7 on Lime:

Report presented in the absence of the chairman by R. S. Boynton, secretary, and the following action taken:

Accepted as Tentative:

Spec. for Fly Ash for Use as a Pozzolanic Material With Lime (C 379 – 56 T)

The report also included a recommendation for the immediate revision of Standard Methods of Sampling, Inspection, Packing, and Marking of Quicklime and Lime Products (C 50 – 27). Since this proposed revision had not been published previously as tentative, it required a nine-tenths vote for approval. The recommendation failed to receive the necessary nine-tenths vote.

Committee C-11 on Gypsum:

Report presented in the absence of the chairman by O. H. Storey, Jr., secretary, and the following actions taken:

Accepted as Tentative:

Spec. for Annular Ringed Nails for Gypsum Wallboard (C 380 – 56 T)

Adopted as Standard, Revisions in:

Methods of Testing Gypsum and Gypsum Products (C 26 – 54)

Committee D-2 on Petroleum Products and Lubricants:

Report presented by H. M. Smith, acting chairman, and the following actions taken:

Accepted as Tentative:

Test for Emulsion Characteristics of Steam-Turbine Oils (D 1401 – 56 T)

Test for Estimation of Heat of Combustion of Liquid Petroleum Products (D 1405 – 56 T)

Test for Effect of Copper on Oxidation Rate of Grease (D 1402 – 56 T)

Test for Cone Penetration of Lubricating Grease Using One-Quarter Scale Cone Equipment (D 1403 – 56 T)

Test for Estimation of Deleterious Particles in Lubricating Grease (D 1404 – 56 T)

Methods for Liquid Calibration of Tanks (D 1406 – 56 T)

Methods for Calibrating Barge Tanks (D 1407 – 56 T)

Methods for Calibrating Stationary Horizontal Tanks (D 1410 – 56 T)

Methods for Calibrating Spherical and Spheroidal Tanks (D 1408 – 56 T)

Methods for Calibrating Tank Car Tanks (D 1409 – 56 T)

Accepted as Tentative, Revisions in:

Test for Saponification Number of Petroleum Products by Color-Indicator Titration (D 94 – 55 T)

Test for Existent Gum in Fuels by Jet Evaporation (D 381 – 54 T)

Spec. for Gasoline (D 439 - 55 T), with the withdrawal of the revision of Section 3; also in the Appendix, Distillation, first paragraph, change the last sentence to read: "These distillation and vapor pressure characteristics define and control: starting, warm-up, carburetor icing, acceleration, vapor lock, crankcase dilution, and in part fuel economy."

Test for Ignition Quality of Diesel Fuels by the Cetane Method (D 613 - 48 T), with the following additional changes:

Footnote 2.—Add the following sentence to footnote 2: "To permit users of the ASTM-CFR Cetane Unit time to acquire this new equipment, these revisions will become officially effective on June 1, 1957."

Section 9 (a).—Add the following at the end of this section:

Now check meter for zero by rotating selector switch to INJECTION ADVANCE position while by-passing fuel to injector. If meter does not read zero, replace tube 6AC7 in the meter. Again rotate selector switch to CALIBRATE and recheck the 25-deg reading.

Supplement III on Operation.—The revision should indicate that Fig. 47 should be deleted (p. 93).

Index.—Add the following to the items listed in the report to be deleted: "Fuel Gap Setting, Cetane, 69."

Supplement VII on Instrumentation.—In Section 703 (*c*), fifth sentence, change "one half turn" to read "between the limits of one-half to one and one-half turns."

In Section 704, following the fourth sentence, add the same material as recommended above for Section 9 (*a*).

In Section 709 (*a*), the second paragraph, beginning with the words "If it is suspected that any one of the pickups is defective . . ." delete the remaining material of this section and replace with the following: "If a pickup is suspected of being faulty, it should be removed from the engine and checked for continuity. If the pickup is either open, short circuited or otherwise defective, it should be repaired or replaced."

In Section 709 (*b*), change the phrase in the note from "50 cycles per minute" to read "50 cycles per second."

Test for Knock Characteristics of Aviation Fuels by the Aviation Method (D 614 - 49 T), with the withdrawal of the proposed revisions of Sections 8(*a*), 8(*b*), and Fig. 6; the deletion of the next to last sentence of the material to be added to Sections 317 and 323 in Supplement III; and the withdrawal of the revisions of Sections 324 (*a*) and 324 (*d*).

Test for Oil Content of Petroleum Waxes (D 721 - 55 T)

Spec. for Aviation Gasolines (D 910 - 53 T)

Test for Olefinic Plus Aromatic Hydrocarbons in Petroleum Distillates (D 1019 - 55 T)

Method of Gaging Petroleum and Petroleum Products (D 1085 - 52 T). Two appendices to these methods were submitted for publication as information.

Method of Measuring the Temperature of Petroleum and Petroleum Products (D 1086 - 54 T)

Test for Reduced Pressure Distillation of Petroleum Products (D 1160 - 52 T)

Methods for Calibrating Upright Tanks (D 1220 - 55 T)

Method for Polarographic Determination of Tetraethyllead in Gasoline (D 1269 - 53 T)

Test for Hydrocarbon Types in Liquid Petroleum Products (Fluorescent Indicator Adsorption (FIA) Method) (D 1319 - 55 T)

Test for Water in Petroleum Products and Other Bituminous Materials (D 95 - 46), subject to concurrence of Committee D-4 on Road and Paving Materials

Adopted as Standard:

Test for Tetraethyllead in Gasoline (D 526 - 53 T)

Test for Evaporation Loss of Lubricating Greases and Oils (D 972 - 51 T)

Test for Mercaptan Sulfur in Jet Fuels (Color-Indicator Method) (D 1219 - 52 T)

Test for Mercaptan Sulfur in Jet Fuels (Amperometric Method) (D 1323 - 54 T)

Test for Copper Corrosion by Petroleum Products (D 130 - 55 T)

Method of Analysis of Oil-Soluble Sodium Petroleum Sulfonates (D 855 - 52 T)

Method of Analysis of Calcium and Barium Petroleum Sulfonates (D 1216 - 52 T)

Adopted as Standard, Revisions in:

Test for Flash Point by Tag Closed Tester (D 56 - 52), jointly with Committee D-1

Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (D 86 - 54)

Test for Viscosity by Means of the Saybolt Viscometer (D 88 - 53)

Test for Flash and Fire Points by Means of Cleveland Open Cup (D 92 - 52)

Test for Vapor Pressure of Petroleum Products (Reid Method) (D 323 - 55)

Test for Knock Characteristics of Motor Fuels by the Motor Method (D 357 - 53)

Test for Knock Characteristics of Motor Fuels by the Research Method (D 908 - 55)

ASTM-IP Petroleum Measurement Tables (D 1250 - 55)

Tentative Withdrawn:

Test for Steam Emulsion of Lubricating Oils (D 157 - 51 T)

Accepted for Publication as Information Only:

Method of Oxidation Test for Turbine Oils (Short-Term Method)
Test for Sulfur in Petroleum Products by the Rapid, High-Temperature Combustion Method
Test for Active Sulfur in Cutting Fluids
Test for Hydrocarbon Types in Gasoline by Mass Spectrometry
Reduction of Observed API Gravity to API Gravity at 60 F—High-Temperature Range
Reduction of Volume to 60 F Against API Gravity at 60 F—High-Temperature Range
Test for Density and Specific Gravity of Viscous Materials and Melted Solids by Bingham Pycnometer Method
Test for Density and Specific Gravity of Hydrocarbon Liquids by Lipkin Bicapillary Viscous Oil Pycnometer
Test for Low-Temperature-Torque of Ball-Bearing Greases
Test for High-Temperature Anti-Friction-Bearing Grease Tester
Reduction of Volume to 60 F Against API Gravity at 60 F—Low-Temperature Range
Reduction of Volume to 60 F Against API Gravity at 60 F—Low-Temperature Range (Abridged Table)
Test for Measuring the Color of Petroleum Products (ASTM Color Scale)
Test for Filterability of Jet Fuels

The committee asked for permission to include four additional proposals as follows:

Proposed Method of Test for Thermal Stability of Fuel Oils
Proposed Extension of Octane Number Scale
Proposed Design and Operation of Automatic Gaging Devices
Proposed Measurement of Tanks Containing Plastic Foam Blankets

The committee withdrew from the report as preprinted the recommendation for the adoption as standard of the Tentative Method of Test for Penetration of Petrolatum (D 937 - 49 T).

Committee D-3 on Gaseous Fuels:

Report presented in the absence of the chairman by P. J. Smith, and the following action taken:

Adopted as Standard:

Test for Sulfur in Fuel Gases (D 1072 - 54 T)

Committee D-8 on Bituminous Waterproofing and Roofing Materials:

Report presented by H. R. Snoke, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Woven Burlap Fabrics Saturated with Bituminous Substances for Use in Waterproofing (D 1327 - 54T)

Adopted as Standard:

Spec. for Asphalt Roll Roofing Surfaced with Mineral Granules (D 249 - 54 T)

Adopted as Standard, Revisions in:

Spec. for Asphalt-Saturated Felt for Use in Waterproofing and in Constructing Built-Up Roofs (D 226 - 47)
Spec. for Coal-Tar Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs (D 227 - 47)
Spec. for Asphalt-Saturated Asbestos Felts for Use in Waterproofing and in Constructing Built-Up Roofs (D 250 - 47)

Committee D-11 on Rubber and Rubber-like Materials:

Report presented by Simon Collier, chairman, and the following actions taken:

Accepted as Tentative:

Method for Tension Testing of Rubber O-Rings (D 1414 - 56 T)[14]
Test for International Standard Hardness of Vulcanized Natural and Synthetic Rubbers (D 1415 - 56 T)
Methods for Chemical Analyses of Synthetic Elastomers (Solid Butadiene-Styrene Copolymers) (D 1416 - 56 T)
Methods for Testing Synthetic Rubber Latices (Butadiene-Styrene Copolymers) (D 1417 - 56 T)
Rec. Practice for Nomenclature for Synthetic Elastomers and Latices (D 1418 - 56 T) with further changes subject to confirming letter ballot of Committee D-11.
Rec. Practice for Description of Types of Styrene Rubbers (SR) (D 1419 - 56 T) with further changes subject to confirming letter ballot of Committee D-11.
Rec. Practice for Description of Types of Styrene Rubber (SR) and Butadiene Rubber (BR) Latices (D 1420 - 56 T)

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[14] In view of negative votes cast in Committee D-11 on the acceptance of this method it was withheld from publication.

Rec. Practice for Interlaboratory Testing of Rubber and Rubber-like Materials (D 1421 - 56 T)

Accepted as Tentative, Revisions in:

Test for Adhesion of Vulcanized Rubber to Metal (D 429 - 55 T)

Methods of Testing Rubber and Thermoplastic Insulated Wire and Cable (D 470 - 54 T)

Spec. and Methods of Test for Latex Foam Rubbers (D 1055 - 54 T)

Spec. and Methods of Test for Sponge and Expanded Cellular Rubber Products (D 1056 - 54 T)

Test for Compressibility and Recovery of Gasket Materials (D 1147 - 53 T)

Adopted as Standard:

Test for Plasticity and Recovery of Rubber and Rubber-like Materials by the Parallel Plate Plastometer (D 926 - 47 T)

Adopted as Standard, Revisions in:

Test for Identation of Rubber by Means of the Pusey and Jones Plastometer (D 531 - 49)

Spec. for Cotton Rubber-Lined Fire Hose for Public and Private Fire Department Use (D 296 - 49), with the deletion of Note 2, and a change in the last sentence of Section 2 (*a*) as follows subject to confirming letter ballot of Committee D-11:

"If agreed between manufacturer and purchaser at the time of the order, the jackets may be made in whole or in part of fibers other than cotton suitable for assuring satisfactory life."

Committee D-15 on Engine Antifreezes:

Report presented in the absence of the chairman by P. J. Smith, and accepted as a report of progress.

Committee D-18 on Soils for Engineering Purposes:

Report presented by E. J. Kilcawley, chairman, and the following actions taken:

Accepted as Tentative:

Test for Water-Soluble Chlorides Present as Admixes in Graded Aggregate Road Mixes (D 1411 - 56 T) (Jointly with Committee D-4)

Accepted as Tentative, Revisions in:

Test for Moisture-Density Relations of Soils (D 698 - 42 T)[15]

The committee withdrew from the report as preprinted the recommendations for publication as tentative of the Method for Soil Investigation and Sampling by Auger Boring, the Method of Thin Walled Tube Sampling of Soils, and the Method for Penetration Testing and Split Spoon Sampling of Soils.

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[15] The revision of Tentative Method D 698 - 42 T was not concurred in by Committee D-4 on Road and Paving Materials, which committee also has jurisdiction over the method, and the method is now being given further consideration in both committees.

EDITORS' NOTE.—The recommendations affecting standards referred to letter ballot were approved when the ballot was canvassed on September 12.

SUMMARY OF PROCEEDINGS OF THE SECOND PACIFIC AREA NATIONAL MEETING

Los Angeles, Calif., Sept. 16–21, 1956

The Second Pacific Area National Meeting of the Society was held at the Statler Hotel in Los Angeles on September 16–21, 1956, with the Northern and Southern California ASTM District Councils cooperating in making arrangements for the meeting. The General Committee on Arrangements was under the Chairmanship of C. M. Wakeman, of the Los Angeles Harbor Department, with:

Vice Chairman, E. O. Bergman, C. F. Braun & Co.
Vice Chairman, T. Parker Dresser, Jr., Abbot A. Hanks, Inc.
Secretary, M. N. Niesley, California Testing Laboratories, Inc.
Treasurer, B. P. Weintz, Consolidated Rock Products Co.

The registered attendance of the meeting was as follows: 1537 members, committee members, and visitors, plus 146 ladies, or a grand total of 1683.

There were 43 technical sessions with 203 papers scheduled for presentation.

Mr. P. James Rich was Chairman of the Technical Program Committee, with many of the sessions sponsored by technical committees of the Society. In addition, there were 7 industrial lucheons held throughout the week with a "President's Luncheon" on Tuesday, September 18. The technical sessions are set forth below with the luncheons being listed separately.

INDUSTRY LUNCHEONS

A number of special luncheons were arranged termed "Industry Luncheons" since they were intended to provide an opportunity for gatherings of those interested in special fields. These together with the listing of the toastmaster and guest speakers are given below.

Petroleum Luncheon Monday, September 17, 12:15 P.M.

Toastmaster: W. T. Gunn, Director, Division of Refining, American Petroleum Institute, New York

Guest Speaker: Howard G. Vesper, Vice President, Standard Oil Co. of California, San Francisco

Subject: "From Pico No. 4 to Petro Chemicals" (The California Oil Story).[1]

Soils Luncheon Tuesday, September 18, 12:15 P.M.

Toastmaster: Paul Baumann, Assistant Chief Engineer, Los Angeles County Flood Control District, Pasadena, Calif.

Guest Speaker: K. B. Woods, Head of School of Civil Engineering, Purdue University, Lafayette, Ind.

Subject: "Soil Explorations for Site Selection and Engineering Design."[2]

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[1] Published in the ASTM Bulletin, No. 220, February, 1957, p. 21.

[2] Included in separate publication *ASTM STP No. 206*.

Wood Luncheon TUESDAY, SEPTEMBER 18, 12:15 P.M.

Toastmaster: Past-President L. J. MARKWARDT, Assistant Director, U. S. Forest Products Laboratory, Madison, Wis.

Guest Speaker: O. HARRY SCHRADER, JR., General Manager, U. S. Plywood Corp., Seattle, Wash.

Subject: "Increasing Integration in the Manufacture of Forest Products."[3]

Railroad Luncheon TUESDAY, SEPTEMBER 18, 12:15 P.M.

Toastmaster: Senior Vice President R. T. KROPF, Vice President and Director of Research, Belding Heminway Co., Inc., New York

Guest Speaker: J. W. CORBETT, Vice President (Systems Operations), Southern Pacific Co., San Francisco

Subject: "Research and Standardization in the Railroad Industry."[4]

Industrial Water Luncheon THURSDAY, SEPTEMBER 20, 12:15 P.M.

Toastmaster: Past President C. H. FELLOWS, Director, Engineering Laboratory and Research Department, The Detroit Edison Co., Detroit, Mich.

Guest Speaker: CLAUDE K. RICE, Coordinator, Gulf Oil Corp., Philadelphia, Pa.

Subject: "Three Is A Vital Number." (The relations of the Public, Government and Industry in the measurement and abatement of stream pollution.)[5]

Cement and Concrete Luncheon THURSDAY, SEPTEMBER 20, 12:15 P.M.

Toastmaster: ROBERT MITCHELL, President, Consolidated Rock Products Co., Los Angeles, Calif.

Guest Speaker: A. ALLAN BATES, Vice President of Research and Development, Portland Cement Association, Chicago, Ill.

Subject: "Research—and Some of Its Relationships to ASTM."

Paint Luncheon THURSDAY, SEPTEMBER 20, 12:15 P.M.

Toastmaster: C. F. PICKETT, U. S. Department of the Army, Aberdeen Proving Ground, Aberdeen, Md.

Guest Speaker: CLYDE L. SMITH, President, Federation of Paint and Varnish Production Clubs, and President, Vi-Cly Industries, Inc., Compton, Calif.

Subject: "The Federation and Its Relation to the Paint Industry."

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[3] Published in the ASTM BULLETIN, No. 219, January, 1957, p. 25.

[4] To be published in the ASTM BULLETIN, No. 222, May, 1957.

[5] Included in separate publication *ASTM STP No. 207.*

PRESIDENT'S NATIONAL LUNCHEON

WEDNESDAY, SEPTEMBER 19, 12:15 P.M.

In honor of President R. A. SCHATZEL, Vice President, and Director of Engineering, Rome Cable Corp., Rome, N. Y.

As a special guest of honor, Lt. Gen. CLARENCE S. IRVINE, Deputy Chief of Staff (Materiel), United States Air Force, will deliver the principal address.

Toastmaster: Past President N. L. MOCHEL, Metallurgical Manager and Consultant, Westinghouse Electric Corp., Philadelphia, Pa.

Introduction of President Schatzel: Senior Vice President R. T. KROPF, Vice President and Director of Research, Belding Heminway Co., Inc., New York

Short Address by President Schatzel:

Subject: "Where Do We Go From Here?" This address is published in the October 1956 issue of the ASTM BULLETIN

Main Address by General Irvine:

Subject: "Super Materials for Super Performance." This address is published in the December 1956 issue of the ASTM BULLETIN

TECHNICAL SESSIONS

SYMPOSIUM ON STEAM TURBINE OILS[6]

MONDAY, SEPTEMBER 17, 9:00 A.M.

Sponsored by Technical Committee C on Turbine Oils of Committee D-2 on Petroleum Products and Lubricants)

Presiding: N. L. Mochel (Past President, ASTM), Manager, Metallurgical Engineering, and Consultant, Westinghouse Electric Corp., Philadelphia, Pa.

G. V. Browning (Chairman, Technical Committee C on Turbine Oils), Large Steam Turbine-Generator Dept., General Electric Co., Schenectady, N.Y.

Organizational and Functional Aspects of Technical Committee C—Section I—C.F. Kottcamp, Gulf Oil Co.

Fire Resistant Turbine Fluids—L. W. Manley, G. H. S. Snyder and N. V. Messina, Socony Mobil Oil Co.

Antiwear Requirements for Navy Turbine Oils—H. F. King, Bureau of Ships, and J. R. Belt, USN Engineering Experiment Station.

Evaluation and Performance of Turbine Oils—G. H. von Fuchs, Consultant, Doble Engineering Co.

PLASTICS

MONDAY, SEPTEMBER 17, 9:00 A.M.

Presiding: C. R. Stock (National Director, ASTM), In Charge, Physical Testing Lab., American Cyanamid Co., Stamford, Conn.

F. W. Reinhart (Chairman Committee D-20 on Plastics), Past President, Soc. of Plastics Engrs., and Chief, Organic Plastics Section, National Bureau of Standards, Wash., D. C.

Correlations Between Electrical Measurements and Mechanical Measurements on Epoxy Plastics Materials—John Delmonte, Furane Plastics, Inc.[7]

Presiding Officers Interlude—"Standards for Plastics—Their Impact on Industry," by Messrs. Stock and Reinhart.

Nondestructive Testing of Bonded Metal Sandwich Materials—Richard Anderson, North American Aviation, Inc.[8]

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[6] Issued as separate publication *ASTM STP No. 211.*

[7] To be published in ASTM BULLETIN.

Included in separate publication *ASTM STP No. 201.*

MISCELLANEOUS

MONDAY, SEPTEMBER 17, 9:00 A.M.

Presiding: R. A. Schatzel (President, ASTM), Vice President and Director of Engineering, Rome Cable Corp., Rome, N. Y.
M. B. Niesley (Chairman, So. Calif. Dist., Secretary West Coast Committee), President, California Testing Labs., Inc., Los Angeles
P. James Rich (Chairman, Technical Program Committee), Consultant

Specifications Should Be Used in Buying Rubber Parts—Raymond B. Stringfield, Consulting Chemical Engineer.[9]

Testing in the Guided Missile Industry—Rebecca H. Sparling, Convair Division, General Dynamic Corp.[10]

Solvent Degreasing—What Every User Should Know—C. E. Kircher, Detrex Corp.[11]

SYMPOSIUM ON TITANIUM[12]

Three Sessions

MONDAY, SEPTEMBER 17, 9:00 A.M.

(*Sponsored by Committee B-2 on Non-Ferrous Metals and Alloys and Administrative Committee on Research*)

Presiding: T. W. Lippert (Chairman, Subcommittee on Titanium), General Manager, Titanium Metals Corp. of America, New York
J. R. Long (Symposium Chairman), Metallurgist, Harvey Machine Co., Torrance, Calif.

Variables Effecting the Thermal Stability of Three Titanium Alloys—F. R. Schwartzberg, N. D. Williams, and R. I. Jaffee, Battelle Memorial Institute.

The Effect of Temperature on the Uniform Elongation of Titanium Alloys—F. C. Holden, H. R. Ogden and R. I. Jaffee, Battelle Memorial Institute.

The Effect of Composition and Annealing Treatment on the Thermal Stability of Ti-Cr-Mo-Alloys—H. R. Ogden, F. C. Holden and R. I. Jaffee, Battelle Memorial Institute.

Elevated Temperature Properties of the Ti-6A-4V Alloy—R.G. Sherman, W. M. Parris and H. D. Kessler, Titanium Metals Co. of America.

Properties and Fabrication Characteristics of Wrought Titanium Products—Leston B. Stark, North American Aviation, Inc.

(Continued at 2:15 P.M. on Monday and 9 A.M. on Tuesday)

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[9] Published in the ASTM BULLETIN, No. 220, February, 1957, p. 39 (TP27).
[10] Published in the ASTM BULLETIN, No. 218, December, 1956, p. 52 (TP230).
[11] Published in the ASTM BULLETIN, No. 219, January, 1957, p. 44 (TP16).
[12] Issued as separate publication *ASTM STP No. 204*.

SYMPOSIUM ON WOOD FOR MARINE USE AND ITS PROTECTION FROM MARINE ORGANISMS[13]

MONDAY, SEPTEMBER 17, 9:00 A.M.

(Sponsored by Committee D-7 on Wood)

Presiding: T. P. Dresser, Jr. (Vice Chairman, General Committee on Arrangements), Chief Engineer, Abbot A. Hanks, Inc., San Francisco, Calif.
C. M. Wakeman (Chairman, General Committee on Arrangements), Testing Engineer, City of Los Angeles Harbor Dept., Los Angeles, Calif.

The Distribution and Importance of Marine Wood Borers in the United States—Robert Menzies, Lamont Geological Observatory, and Ruth Turner, Museum of Comparative Zoology, Harvard University.

Marine Exposure Tests of Wood Treated with Various Preservatives—A.P. Richards, William F. Clapp Laboratories, Inc.

Performance Tests of Heavy Metal Compounds as Marine Borer Inhibitors—T. Roe, Jr., H. Hochman and E. R. Holden, U. S. Naval Civil Engineering Research and Evaluation Laboratory.

The Importance of the Local Borer Species in Specifying Agents for Protecting Wood from Marine Borers—Harold Vind, Jim Maraoka, Joan Casey and H. Hochman, U. S. Naval Civil Engineering Research and Evaluation Laboratory.

The Migration of Marine Borers—C. H. Edmondson, Bernice P. Bishop Museum. (Not published)

SYMPOSIUM ON STEAM TURBINE OILS (*Continued*)[6]

MONDAY, SEPTEMBER 17, 2:15 P.M.

Presiding: E. W. Adams (Secretary, Technical Committee C on Turbine Oils), Associate Director of Research, Standard Oil Co. of Indiana

Practices for Determining the Expected Life of Used Turbine Oils—R. G. Mastin, Cities Service Oil Co.

Rusting in Turbine Oil-Systems—N. W. Furby, F. J. Hanly, California Research Corp., and J. A. Vincent, Standard Oil Company of California.

Monolayers of Anti-Rust Additives—H. E. Ries, H. D. Cook and C. M. Loane, Standard Oil Co. Ind.

Water Leaching of Additives—E. W. Brennan and R. G. Moyer, The Pure Oil Co.

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[13] Issued as separate publication *ASTM STP No. 200.*

ROAD AND PAVING MATERIALS[14]

MONDAY, SEPTEMBER 17, 2:15 P.M.

(Sponsored by Committee D-4 on Road and Paving Materials)

Presiding: B. A. Vallerga, Managing Engr., Pacific Coast Region, The Asphalt Institute, San Francisco, Calif.

F. N. Hveem (Member, Program Committee), Materials and Research Engineer, Calif. Div. of Highways, Sacramento

Effect of Shape, Size and Surface Roughness of Aggregate Particles on the Strength of Granular Materials—B. A. Vallerga, H. B. Seed, C. L. Monismith and R. S. Cooper, University of California.

The Operation, Control and Application of the Infrared Weathering Machine—California Design—John B. Skog, California Division of Highways.

Correlation Between Laboratories on Results of ASTM Tests for Asphalts—Ernest Zube and John B. Skog, California Division of Highways.

Sliding Plate Microviscometer for Rapid Measurement of Asphalt Viscosity in Absolute Units—R. L. Griffin, T. K. Miles, C. J. Penther and W. C. Simpson, Shell Development Co.

Reproducibility in Oven Heat Tests for Paving Asphalts—R. S. Winniford, California Research Corp.

FERROUS METALS[15]

MONDAY, SEPTEMBER 17, 2:15 P.M.

Presiding: C. R. Baker (Vice Chairman, Luncheons Committee), Progress Engineer, Pacific Tube Co., Los Angeles, Calif.

L. H. Winkler (Honorary Member), Metallurgical Engineer, Bethlehem Steel Co., Inc., Bethlehem, Pa.

F. S. Jacobson (Member, Program Committee), Metallurgical Engineer, Research & Development, Kaiser Steel Corp., Fontana, Calif.

Shotpeening Effects and Specifications—Henry O. Fuchs, Metal Improvement Co.

Special Techniques for Increasing Strength and Fatigue Life of Steel Stressed in Torsion—N. E. Hendrickson, Rheem Automotive Co.[16]

The Isothermal Evolution of Hydrogen from Arc Welds in Mild Steel—Alan E. Flanigan and Eun U. Lee, University of California. (Not published)

Studies of Stainless Steel Columns Subject to Compression Loads—Julien Dubuc and Georges Welter, Ecole Polytechnique, and V. N. Krivobok, The International Nickel Co., Inc.

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[14] Issued as separate publication *ASTM STP No. 212.*

[15] Issued as separate publication *ASTM STP No. 196.*

[16] To be published in ASTM BULLETIN.

NONDESTRUCTIVE TESTING[17]

Report on Activities of Committee E-7

MONDAY, SEPTEMBER 17, 2:15 P.M.

Presiding: J. H. Bly (Chairman, Committee E-7), Sales Div., High Voltage Engineering Corp., Cambridge, Mass.

T. A. Triplett (Member Exec. Council E-7), President, Triplett & Barton, Inc., Burbank, Calif.

G. H. Tenney, (Member, Committee E-7) Groupleader, Los Alamos Scientific Lab., Los Alamos, N. Mex.

Introduction—Committee E-7 Operation—J. H. Bly, High Voltage Engineering Corp.

Recommended Practices for Radiographic Inspection—E. A. Burrill, High Voltage Engineering Corp.

Magnetic Particle and Penetrant Testing Procedures—Hamilton Migel, Magnaflux Corp.

Reference Radiographs for Inspection Purposes—N. A. Kahn, New York Naval Shipyard.

Radiographic Procedures—C. H. Hastings, Watertown Arsenal.

Report on the Activities and Future Projects of the Ultrasonic Subcommittee VI of Committee E-7—John Smack, Curtiss-Wright Corp.

SYMPOSIUM ON TITANIUM[12]

MONDAY, SEPTEMBER 17, 2:15 P.M.

Presiding: R. E. Paine (Vice Chairman, So. Calif. Dist.), Chief Metallurgist, Aluminum Company of America, Los Angeles, Calif.

B. W. Gonser (Chairman, Committee B-2 on Non-Ferrous Metals and Alloys), Assistant Director, Battelle Memorial Institute, Columbus, Ohio

A Micro Notched-Bar Impact Test for Titanium Alloys—F. C. Holden, H. R. Ogden and R. I. Jaffee, Battelle Memorial Institute.

The Measurement of Elastic Modulus of Titanium Alloys—W. H. Graft and W. Rostoker, Armour Research Foundation.

Ignition and Stress Corrosion Cracking of Titanium in Fuming Nitric Acid—John B. Rittenhouse, Jet Propulsion Laboratory.

Unalloyed Titanium Sheet Is Improving—Leo Schapiro and H. A. Barry, Douglas Aircraft Co., Inc.

How Statistical Techniques Helped Achieve Better Uniformity in Unalloyed Titanium—Chester R. Smith, Mallory-Sharon Titanium Corp.

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[17] Issued as separate publication *ASTM STP No. 213.*

SYMPOSIUM ON RAILROAD MATERIALS[18]

Two Sessions

TUESDAY, SEPTEMBER 18, 9:00 A.M.

Part 1: Diesel Fuels and Lubricants

Presiding: P. V. Garin (National Director, ASTM, Symposium Chairman), Engineer of Research & Mechanical Standards, Southern Pacific Co., San Francisco, Calif.
W. F. Collins (Chairman, Committee A-1 on Steel), Asst. Chief, Engineering Services, New York Central System, New York City
M. A. Pinney (Member, Administrative Committee on Standards), Engineer of Tests, The Pennsylvania Railroad Co., Altoona, Pa.

Operation of Railroad Diesel Locomotives with Dual Fuel Systems—P. V. Garin, Southern Pacific Co.

Locomotive Lubricating Oil Requirements as Related to Fuels—J. L. Broughten and C. C. Moore, Union Oil Co. of California.

Performance of Residual Fuels in High Speed Diesel Engines—D. R. Jones, K. L. Kipp and J. E. Goodrich, California Research Corp.

Laboratory Research on Burning No. 6 Residual Fuel in an 8⅛ x 10 Opposed Piston Diesel Engine—R. H. Beadle, Fairbanks, Morse & Co.

SOILS[19]

Three Sessions

TUESDAY, SEPTEMBER 18, 9:00 A.M.

(Sponsored by Committee D-18 on Soils for Engineering Purposes)

Presiding: K. B. Woods, Jr. (Vice President, ASTM), Head, School of Civil Engineering, Purdue Univ., Lafayette, Ind.
F. J. Converse (Symposium Chairman and 2nd Vice Chairman Committee D-18), Professor of Soil Mechanics, Calif. Institute of Tech., Pasadena
R. Horonjeff, Institute of Transportation & Traffic Engineering, University of Calif., Berkeley

Soil Density and Soil Moisture Determination by Radiation Methods—R. K. Bernhard, Rutgers University.

Field Tests on Laterally Loaded Instrumented Piles—"Fixed Head" Loadings in Sand—"Free Head" Loadings in Clay—Harold G. Mason, U. S. Naval Civil Engineering Research Laboratory.

A Generalized Theory of Soil Resistance—W. S. Housel, University of Michigan.

A Procedure for Separately Evaluating Friction and Cohesion by a Consolidated Direct Shear Test—L. A. Palmer, Philip B. Brown and C. M. Yeomans, U. S. Navy Bureau of Yards & Docks.

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[18] Issued as separate publication *ASTM STP No. 214*.

[19] Issued as separate publication *ASTM STP No. 206*.

SYMPOSIUM ON WOOD POLES

TUESDAY, SEPTEMBER 18, 9:00 A.M.

(Sponsored by Committee D-7 on Wood)

Presiding: F. B. Doolittle (Chairman, Plant Visits Committee), Electrical Engineer, Southern Calif. Edison Co., Los Angeles

L. J. Markwardt (Past President ASTM, Symposium Chairman), Asst. Dir., U. S. Forest Prods. Lab., Madison, Wis.

Wood Poles for Communication Lines—G. Q. Lumsden, Bell Telephone Laboratories, Inc.[20]

A Look Ahead at Wood Pole Production in Relation to the Forest Resource—E. E. Matson, Forest Utilization Service.

Need for Research on Wood Poles—L. J. Markwardt, U. S. Forest Products Laboratory.

Testing Poles for the American Society for Testing Materials—L. W. Wood, U. S. Forest Products Laboratory.

Engineering of Specifications for Wood Poles—L. G. Smith, Baltimore Gas & Electric Co.[21]

SYMPOSIUM ON NONDESTRUCTIVE TESTING[17]

Two Sessions

TUESDAY, SEPTEMBER 18, 9:00 A.M.

(Sponsored by Committee E-7 on Nondestructive Testing)

Presiding: L. P. Spalding (Past National Director, ASTM), Director, Laboratory, North American Aviation, Inc., Los Angeles, Calif.

W. C. Hitt (Symposium Chairman), Asst. Chief, Quality Control, Douglas Aircraft Co., Inc., Santa Monica, Calif.

Development of Technique for the Detection of Cracks in Small Cylindrical Specimens by Eddy Current and Reluctance Methods—Eugene Roffman, Frankford Arsenal.

Electronic X-Ray Image Systems—Richard F. Holste, J. E. Jacobs, and A. Pace, General Electric Co.

Advanced Ultrasonic Testing in Europe and America—Donald Erdman, Electro Circuits, Inc.

Recent Developments in the Use of Ultrasonic Attenuation and Velocity Measurements in the Study of Properties of Materials—John Truell, Brown University.

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[20] To be published in the ASTM BULLETIN, No. 222, May, 1957.

[21] Published in *Electric Light and Power.*

SYMPOSIUM ON TITANIUM[12]

TUESDAY, SEPTEMBER 18, 9:00 A.m.

Presiding: F. L. LaQue (National Director, ASTM), Vice President & Manager, Development & Research Div., The International Nickel Co., Inc., New York
F. T. Wood, Jr., Chief, Materials & Process Engineer, Douglas Aircraft Co., Santa Monica, Calif.

Development of Standardized Sample, Preparation and Testing Techniques for Unalloyed Titanium Sheet—R. L. Folkman and M. Schussler, Electro Metallurgical Co.

Oxygen Analysis—R. M. Fowler, Electro Metallurgical Co.

Titanium Sponge Quality Evaluation at Titanium Metals Corporation of America—Carl Blake and Lawrence T. Eck, Titanium Metal Co. of America. (Not published)

Observation on Brinell Hardness Testing of Titanium Sponge—Don Baker, Bureau of Mines. (Not published)

Development of Titanium-Base Alloys for Elevated Temperature Application. Part 1: Binary Alloys; Part II: Ternary Alloys; Part III: Quaternary Alloys—F. A. Crossley, W. F. Carew, Armour Research Foundation, and H. D. Kessler, Titanium Metals Corp., (Formerly with Armour Research Foundation). Presented by title only.

SOILS (*Continued*)[19]

TUESDAY, SEPTEMBER 18, 2:15 P.M.

Presiding: J. B. Howe (Chairman, Publicity Committee), Associate and Field Engineer Maurseth & Howe, Los Angeles
W. S. Housel, (First Vice Chairman, Committee D-18), Assoc. Prof. Civil Engineering, Univ. of Michigan, Ann Arbor

Experiences with Consolidation of Pipe Bedding by Vibration on the San Diego Aqueduct—W. G. Holtz, U. S. Bureau of Reclamation.

The Use of Vibratory Compactors on Granular Base Courses—C. R. White, U. S. Naval Civil Engineering Research Laboratory.

Vibratory Compaction of Cohesive Soil by Low Frequency Vibration—F. J. Converse, California Institute of Technology.

Microseismics—R. K. Bernhard, Rutgers University.

SYMPOSIUM ON LUBRICATING OILS[18]

TUESDAY, SEPTEMBER 18, 2:15 P.M.

(Sponsored by Research Division III on Elemental Analysis and Technical Committee B on Lubricating Oils of Committee D-2 on Petroleum Products and Lubricants)

Part I: Spectrographic Analysis of Lubricating Oils

Presiding: C. E. Emmons (West Coast Committee), Retired. Formerly Regional Manager, Technical Service, The Texas Co., Los Angeles. C. M. Gambrill (Chairman, Research Division on Elemental Analysis), Mgr., Analytical Research & Service Labs., Ethyl Corp., Detroit, Mich.

R. L. LeTourneau (Symposium Chairman), California Research Corp., San Francisco, Calif.

Introduction—C. M. Gambrill, Ethyl Corp.

Direct-Reading Spectrographic Control of Lubricating Oil Additive Manufacture—R. E. Ramsay, California Research Corp., and W. A. Rappold, Oronite Chemical Co.

The Application of X-Ray Spectrography to Refinery Control of Additive Metals in Lubricating Oils—E. N. Davis, Sinclair Research Laboratories, Inc.

Sampling of Lubricating Oil from Diesel Locomotives—W. K. Simpson, General Motors Corp., Electro-Motive Engineering Division.

Section J Committee Report on Spectrographic Analysis of Lubricating Oils—Ford R. Bryan, Ford Motor Co.

Direct Determination of Metals in Used Lubricating Oils by Emission Spectrography—W. D. Perkins, J. R. Miller, and J. H. Moser, Shell Oil Co.

Direct Reading Spectrographic Evaluation of Used Railroad Oils—V. C. Barth, Chicago and Northwestern Railway System.

Part II: General Papers

Presiding: D. E. Bowers (Member, West Coast Committee), General Petroleum Corp., Los Angeles, Calif.

W. S. James (Chairman, Technical Committee B on Lubricating Oils), William S. James & Associates, Birmingham, Mich.

J. B. Gregory (Chairman, Subcommittee on Elements in Lubricants), Union Oil Co., Brea, Calif.

Introduction—W. S. James, William S. James & Associates.

Unsolved Problems in Analysis of Diesel Lubricating Oils—W. E. Lasky, Gulf Mobile and Ohio Railroad.

Chromatography of Lubricating Oils for Diesel Locomotives—E. R. Thomas, Southern Pacific Railroad.

Filtering Diesel Engine Lubricating Oils—S. L. Earle, U. S. Naval Experiment Station.

SYMPOSIUM ON FULL-SCALE TESTS ON HOUSE STRUCTURES[22]

TUESDAY, SEPTEMBER 18, 2:15 P.M.

(Sponsored by Committee E-6 on Methods of Testing Building Constructions and Committee D-7 on Wood)

Presiding: T. K. May (Member, West Coast Committee), Director of Technical Service, West Coast Lumbermen's Association, Portland, Oregon

L. J. Markwardt (Past President, ASTM) Asst. Director, U. S. Forest Products Laboratory, Madison, Wis.

R. F. Leggett (Chairman, Committee E-6 Symposium Chairman), Director of Building Research, N.R.C. of Canada, Ottawa

Rigidity and Strength of Houses Built of Plywood Stressed-Cover Panels—R. F. Luxford, and E. C. O. Erickson, U. S. Forest Products Laboratories.

NAVCERELAB Facilities for Evaluating Prefabricated Buildings—J. E. Dykins, U. S. Naval Research Establishment.

Full Scale Testing of Prefabricated Military Buildings—R. F. Bartelmes, U. S. Corps of Engineers, Research and Development Laboratories.

Structural Test of a House under Simulated Wind and Snow Loads—D. B. Dorey and W. R. Schriever, National Research Council of Canada.

Full-Scale Tests of Pre-Cast Multi-Story Flat Constructions—A. J. Francis, W. P. Brown, and S. Aroni, University of Melbourne.

SYMPOSIUM ON NONDESTRUCTIVE TESTING (*Continued*)[17]

TUESDAY, SEPTEMBER 18, 2:15 P.M.

Presiding: J. C. Smack (Chairman, Subcommittee on Ultrasonic Testing E-7), Sales Staff Engineer, Curtiss-Wright Corp., Caldwell, N. J.

Hamilton Migel (Chairman Subcommittee on Magnetic Particle & Penetrant Testing E-7), Vice President, Engineering, Magnaflux Corp., Chicago, Ill.

Nondestructive Testing on Southern Pacific Railroad—Arthur S. Pedrick, Southern Pacific Co.

Nondestructive Testing of Heavy Metals—Gerold H. Tenney, Los Alamos Scientific Laboratory.

Ultrasonic Inspections of Aircraft Forgings—Alex Barath, Douglas Aircraft Co., Inc.

An Ultrasonic Technique for Non-Destructive Evaluation of Metal-to-Metal Adhesive Bonds—James S. Arnold, Stanford Research Institute.

Ultrasonic Reflectoscope Calibration Techniques—H. E. VanValkenburg, Sperry Products, Inc.

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[22] Issued as separate publication *ASTM STP No. 210*. For the list of papers included in this Symposium, see Summary of Proceedings of the Buffalo Spring Meeting, p. 54.

SYMPOSIUM ON PROPERTIES, TESTS AND PERFORMANCE OF ELECTRODEPOSITED METALLIC COATINGS[23]

Two Sessions

WEDNESDAY, SEPTEMBER 19, 9:00 A.M.

(Sponsored by Committee B-8 on Electrodeposited Metallic Coatings)

Presiding: P. James Rich (Chairman, Technical Program Committee), Consultant
W. L. Pinner (Past President AES, Symposium Chairman), Houdaille Industries, Inc., Detroit, Mich.

(Continued at 2:15 P.M.)

SOILS (*Continued*)[19]

WEDNESDAY, SEPTEMBER 19, 9:00 A.M.

Presiding: John Bishop (Chairman, Soil Mechanics Group L. A. Sect ASCE), Director, Soils & Pavement Div. Research Dept., U. S. N. Civil Engineering Research & Evaluation Lab., Port Hueneme, Calif.
W. G. Holtz (Secretary, Committee D-18), Head, Earth Materials Lab., U. S. Bureau of Reclamation, Denver Federal Center, Denver, Colo.

Strength and Elastic Properties of Soil-Cement Mixtures—Earl J. Felt and Melvin S. Abrams, Portland Cement Assn.

Universal Soil Testing Machine—John W. Maloney, Dames & Moore.

Experiences with Electrical Resistivity Surveys on Foundation and Subsurface Investigations in Southern California—Ray A. Maurseth and Stanley N. Mitchell, Maurseth & Howe.

Tests of Deadman Anchorages in Sand—J. E. Smith, U. S. Naval Civil Engineering Research Laboratory.

SYMPOSIUM ON BUILDING DESIGN FOR SEISMIC AND SHOCK LOADING[24]

WEDNESDAY, SEPTEMBER 19, 9:00 A.M.

(Sponsored by Committees D-7 on Wood and E-6 on Methods of Testing Building Constructions)

Presiding: Ernst Maag (Member of West Coast Committee), Principal Structural Engineer, Division of Architecture, Calif. State Dept. of Public Works, Sacramento
Ben Benioff, Consulting Structural Engineer, Sherman Oaks, Calif.

Building Design for Lateral Forces—H. J. Degenkolb, John J. Gould & H. J. Degenkolb.

Report on Recent Oregon Forest Products Laboratory Tests of Cantilevered Wood Mullions Fixed in Diagonally Sheathed and Plywood Panels—Charles Peterson, California State Division of Architecture.

Report on U. S. Forest Products Laboratory Tests of Full-Size Structural Wood Diaphragms Made in Cooperation with State of California and U. S. Army Engineers—R. P. A. Johnson, U. S. Forest Products Laboratory.

Trends in School Building Design—Harry W. Bolin, Structural Engineering Consultant.[25(a)]

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[23] Issued as separate publication *ASTM STP No. 197*, see page 54 for list of Papers.

[24] Issued as separate publication *ASTM STP No. 209*.

SYMPOSIUM ON RAILROAD MATERIALS (*Continued*)[18]

Wednesday, September 19, 9:00 A.M.

Part II: Diesel Fuels and Lubricants: Cleaning Materials

Presiding: L. S. Crane (Chairman, Committee A-2 on Wrought Iron), Engineer of Tests, Southern Railway System, Alexandria, Va.
E. B. Fields, Engineering & Research, Atchison, Topeka & Santa Fe Railway, Chicago, Ill.

Coordination of Research on Locomotive Diesel Fuels and Lubrication Oils—R. W. Seniff, Baltimore & Ohio Railroad.

Development of Additives for Economy Type Locomotive Diesel Fuels—G. C. Reinhard, XZIT Chemical Co.

Railroad Cleaners and Cleaning Procedures—J. L. Ramsey, Wyandotte Chemical Co.

Standardization of Railroad Cleaning Materials and Methods—C. F. Jursch, Southern Pacific Co.

MASONRY[25]

Wednesday, September 19, 9:00 A.M.

Presiding: R. W. Harrington (Vice Chairman, No. Calif. Dist.) Manager, Clay Brick & Tile Assn., San Francisco, Calif.

Tests of Reinforced-Concrete Masonry Beams—Albyn Mackintosh, Consulting Engineer.

Importance of Testing to the Clay Pipe Industry—A. Lee Bennett, Pacific Clay Products Research Laboratories.

A Test Method for Producing Blast Loading on Clay Masonry Walls—Paul V. Johnson, Structural Clay Products Research Foundation.

Efficient Sampling Techniques in the Ceramic Industry—Glenn D. James, Gladding, McBean & Co.

Research Testing Methods of Plain Grouted, and Reinforced Grouted Brick Masonry—J. Morley English, University of California of Los Angeles, and R. R. Schneider, University of Southern California.

Testing Ceramic Tile—J. Vincent Fitzgerald and E. L. Kastenbein, Tile Council of America, School of Ceramics, Rutgers University.

Effects of Type, Thickness and Age of Capping Compounds on the Apparent Compressive Strength of Brick—Norman W. Kelch, Associated Brick Manufacturers of Southern California, and Frank E. Emme, Raymond G. Osborne Laboratories.

Testing Brick, Mortar and Grout—Color Film with Narration—Robert W. Harrington, Clay Brick & Tile Manufacturers Assn. (Not published)

SYMPOSIUM ON PROPERTIES, TESTS AND PERFORMANCE OF ELECTRODEPOSITED METALLIC COATINGS (*Continued*)[23]

Wednesday, September 19, 2:15 P.M.

Presiding: R. B. Saltonstall (Secretary, Committee B-8), Director of Research, The Udylite Corp., Detroit, Mich.

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[25] Publication undetermined.

SYMPOSIUM ON DEVELOPMENTS IN GLUED-LAMINATED AND OTHER WOOD CONSTRUCTIONS[24]

WEDNESDAY, SEPTEMBER 19, 2:15 P.M.

(Sponsored by Committees D-7 on Wood and E-6 on Methods of Testing Building Constructions)

Presiding: D. M. Wilson (West Coast Committee Member), Prof. Civil Engineering, Univ. So. Calif., Los Angeles

L. J. Markwardt (Past President ASTM, Symposium Chairman), Asst. Director, U. S. Forest Products Laboratory, Madison, Wis.

F. J. Hanrahan (Chairman, Subcommittee Laminated Timber), Exec. Vice President American Institute of Timber Constr., Wash., D. C.

Developments in Glued-Laminated Construction—Robert E. Eby, Rilco Laminated Products, Inc.

Factors Affecting Strength and Design Principles of Glued-Laminated Construction—Alan B. Freas, U. S. Forest Products Laboratory.

Range in Strength Qualities of Dimension Lumber—L. W. Wood, U. S. Forest Products Laboratory.

Developments in Engineered Wood Design and Construction (Other Than Glued-Laminated)—Verne Ketchum, Timber Structures, Inc.[25(b)]

Developments in Softwood Plywood Design and Construction—David Countryman, Douglas Fir Plywood Assn.

SYMPOSIUM ON VAPOR PHASE OXIDATION OF GASOLINE[26]

WEDNESDAY, SEPTEMBER 19, 2:15 P.M.

(Sponsored by Technical Committee on Gasoline of Committee D-2 on Petroleum Products and Lubricants)

Presiding: J. M. Campbell (National Director, ASTM), Technical Dir., Research Labs. Div., General Motors Corp., Detroit, Mich.

R. C. Alden (National Director and Vice Chairman, Committee D-2), Chairman, Research Planning Board, Phillips Petroleum Co., Bartlesville, Okla.

W. R. Power (Symposium Chairman), Asst. Chief Chemist, Cities Service Oil Co., East Chicago, Ind.

Fuel Factors Influencing Intake System Deposition—C. R. Bauer and H. J. Scheule, E. I. du Pont de Nemours & Co., Inc.

Induction System Gum—Engine Versus Bench Test—J. L. Keller and F. S. Leggett, Union Oil Company of California.

Effect of Fuel Composition upon Intake Valve Deposit Forming Characteristics—A. V. Cabal and J. Capowski, Socony Mobil Oil Co., Inc.

Induction System Reactions—Liquid or Vapor?—A. C. Nixon, H. B. Minor and T. P. Rudy, Shell Development Co.

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[26] Issued as separate publication *ASTM STP No. 202.*

CEMENT[27]

WEDNESDAY, SEPTEMBER 19, 2:15 P.M.

(Sponsored by Committee C-1 on Cement)

Presiding: W. C. Hanna (Honorary Member ASTM, Symposium Chairman), Vice President, California Portland Cement Co., Colton, Calif.

R. R. Litehiser (National Director, Chairman Committee C-1), Chief Engineer, Ohio State Highway Testing Lab., Ohio State Univ., Columbus, Ohio

M. A. Swayze (Past National Director), Dir. of Research, Lone Star Cement Corp., New York

Alkali Aggregate Phase of Chemical Reactivity in Concrete, Part II—J. A. Hester and O. F. Smith, Bureau of Materials and Tests, Alabama State Highway Department.

Effect of Storage on Air-Entraining Cements—W. J. McCoy and S. B. Helms, Lehigh Portland Cement Co.

Hydrophobic Cement—U. W. Stoll, U. S. Navy, CERE Lab.

Carbonation of Hydrated Portland Cement—George Verbeck, Portland Cement Assn.

Determination of Magnesium Oxide in Portland Cement by Flame Photometry—L. R. Pritchard, Lone Star Cement Corp.

SYMPOSIUM ON STRUCTURAL SANDWICH CONSTRUCTION[28]

Two Sessions

THURSDAY, SEPTEMBER 20, 9:00 A.M.

(Sponsored by Committee C-19 on Structural Sandwich Constructions)

Presiding: G. R. Little (Member, West Coast Committee), Chief Engineering Inspector, City of Los Angeles, Dept. of Water & Power, Los Angeles, Calif.

L. J. Markwardt (Past President ASTM), Asst. Dir., U. S. Forest Products Lab., Madison, Wis.

E. W. Kuenzi (Past-Chairman, Committee C-19), Civil Engineer, U. S. Forest Prods. Lab., Madison, Wis.

High Temperature Testing of Adhesives for Aircraft Structural Application—J. R. Battalora and D. E Pulsifer, Kawneer Co.

Effect of Dimensional Factors and Temperature on the Shear Strength of Aluminum Honeycomb—W. Cheorvas, North American Aviation, Inc., and W. C. Plumtree, Los Angeles City College.

Sandwich in the Design of Helicopters—J. M. Stevens, U. S. Dept. of the Navy, Bureau of Aeronautics.

Conclusions Derived from Empirical Studies of Bonded Details for Sandwich Construction—M. L. Sheridan and H. R. Merriman, Glenn L. Martin Co.

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[27] Issued as separate publication *ASTM STP No. 205.*

[28] Issued as separate publication *ASTM STP No. 201.*

SYMPOSIUM ON RADIATION EFFECTS ON MATERIALS[29]

Two Sessions

THURSDAY, SEPTEMBER 20, 9:00 A.M.

(Sponsored by Subcommittee on Radiation Effects of Atomic Industrial Forum and ASTM Committee E-10 on Radioactive Isotopes)

Presiding: R. W. Truesdail (Secretary, American Council of Independent Laboratories), President, Truesdail Labs., Inc., Los Angeles, Calif.

C. C. Woolsey (Member, Special Committee Nuclear Problems), Group Leader, Fuel Element Development, Atomics International Div., North America Aviation, Inc., Canoga Park, Calif.

D. O. Leeser (Symposium Chairman), Atomic Power Development Associates, Inc., Detroit, Mich.

I. Theory

Displaced Atoms in Solids—Comparison Between Theory and Experiment—G. J Dienes, Brookhaven National Laboratory.

II. Radiation Facilities and Mechanics of Testing

A Radiation Effects Program—J. E. Whitney, E. M. Chandler, J. E. Gates, and G. D. Calkins, Battelle Memorial Institute.

The Mechanics of Testing Irradiated Materials—R. G. Berggren, C. Dismuke, M. J. Feldman, and J. C. Wilson, Oak Ridge National Laboratory.

Westinghouse Testing Reactor—A. W. De Agazio, Westinghouse Electric Corp.

III. Experimental—A. Fuel and Graphite Materials

Survey of Radiation Effects on Fuel Materials—Dwain Bowen, North American Aviation, Inc.

Influence of Heat Treatment on Irradiation-Induced Dimensional Changes in Some Uranium-Zirconium Alloys—J. H. Kittel, S. H. Paine, and H. H. Chiswik, Argonne National laboratory.

Radiation Effects on Graphite—R. L. Carter, Atomics International, North American Aviation, Inc.

NON FERROUS METALS[15]
INCLUDING 1956 GILLETT LECTURE

THURSDAY, SEPTEMBER 20, 9:00 A.M.

Presiding: R. A. Schatzel (President ASTM), Vice Pres. & Director of Engineering. Rome Cable Corp., Rome, N. Y.

G. H. Harnden (National Director, Chairman Committee B-5), Consultant, Materials & Processes, Engineering Services Div., General Electric Co., Schenectady

R. E. Paine (Vice Chairman So. Calif. Dist.), Chief Metallurgist, Aluminum Company of America, Los Angeles, Calif.

Condensed Version of the 1956 Gillett Lecture on Structural Chemistry and Metallurgy of Copper—D. K. Crampton, Chase Brass and Copper Co. (Presented by Henry Burghoff, Metallurgist, Chase Brass and Copper Co.)[30]

Pit Depth Measurements as a Means of Evaluating the Corrosion Resistance of Aluminum in Sea Water—T. J. Summerson, M. J. Pryor, D. S. Keir, and R. J. Hogan, Kaiser Aluminum & Chemical Corp.

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[29] Issued as separate publication *ASTM STP No. 208.*

[30] The Gillett Lecture is published only as a separate pamphlet and not included in *ASTM STP No. 196.*

The Properties of Beryllium Copper Strip as Affected by Cold Rolling and Heat Treating—J. T. Richards and Ellsworth M. Smith, Penn Precision Products, Inc.

Determination of Young's Modulus Under Conditions of Relaxation—R. W. Fenn, Jr., The Dow Chemical Co.

CONCRETE[27]

THURSDAY, SEPTEMBER 20, 9:00 A.M.

(Sponsored by Committee C-9 on Concrete and Concrete Aggregates)

Presiding: E. O. Slater (Past District Chairman; Past National Director), President, Smith-Emery Co., Los Angeles, Calif.

R. E. Davis (Vice Chairman, Technical Program Committee, Symposium Chairman), Director Emeritus, Engineering Materials Lab., Univ. of Calif., Berkeley

Byron Weintz (Chairman, Finance Committee, Vice Chairman, So. Calif. Dist.). Chief Engineer, Consolidated Rock Products Co., Los Angeles

The Partial Replacement of Portland Cement in Concrete—Bryant Mather, Waterways Experiment Station, Corps of Engineers.

Test of Prestressed Expanded Shale Concrete Beams Subject to Short-Time and Sustained Loads—R. E. Davis and G. E. Troxell, University of California, Don McCall, and R. A. McCann, Basalt Rock Co.

The Effect of Aggregate Quality on Resistance of Concrete to Abrasion—Fabian L. Smith, U. S. Bureau of Reclamation.

A Physical Method for Determining the Composition of Hardened Concrete—J. W. Kelly, Milos Polivka, and C. H. Best, University of California.

SYMPOSIUM ON INDUSTRIAL WATER AND INDUSTRIAL WASTE WATER[31]

Two Sessions

THURSDAY, SEPTEMBER 20, 9:00 A.M.

(Sponsored by Committee D-19 on Industrial Water)

Presiding: C. H. Fellows (Past President ASTM), Director, Engineering Lab & Research Dept., The Detroit Edison Co., Detroit, Mich.

L. D. Betz (Symposium Chairman), Gen. Manager, W. H. & L. D. Betz, Phila, Pa.

Industrial Waste Problems in Southern California—T. C. Wilson, Los Angeles Bureau of Standards.

Water Pollution Control in the Los Angeles Area—Carl B. Johnston, Pomeroy and Associates (Formerly Los Angeles Regional Water Pollution Control Board No. 4.)

Committee D-19: The First Quarter Century—Robert C. Adams, U. S. Naval Engineering Experiment Station.

THURSDAY, SEPTEMBER 20, 2:15 P.M.

Presiding: R. C. Adams (Vice Chairman, Committee D-19), Supt., Chemical Engineering Lab., U. S. N. Engineering Experiment Sta., Annapolis, Md.

Sea Water Purification—O. M. Elliott, Sun Oil Co.

The Use of Organic Flocculants and Flocculating Aids in the Treatment of Industrial Water and Industrial Waste Water—J. K. Rice, Cyrus Wm. Rice & Co.

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[31] Issued as separate publication *ASTM STP No. 207.*

SYMPOSIUM ON RADIATION EFFECTS ON MATERIALS (*Continued*)[29]

THURSDAY, SEPTEMBER 20, 2:15 P.M.

II Experimental—B. Structural Materials Including Organics

Presiding: Frank Linn (Member, Special Committee Nuclear Problems), Aircraft Nuclear Propulsion Div., General Electric Co., Evendale, Ohio

G. D. Calkins (Honorary Chairman, Committee E-10), Battelle Memorial Institute, Columbus, Ohio

J. B. Austin (Atomic Industrial Forum), Vice Pres., Fundamental Research, U. S. Steel Corp., Pittsburgh, Pa.

Survey of the Effects of Neutron Bombardment on Structural Materials—L. Castleman, Atomic Energy Division, Sylvania Electric Products.

A Summary of the Effect of Irradiation on Some Plastics and Elastomers—Oscar Sisman and C. D. Bopp, Oak Ridge National Laboratory.

Effect of Irradiation on the Notched-Bar Impact Properties of Some Plain-Carbon Steels—D. O. Leeser, Atomic Power Development Associates, Inc. and G. J. Deily, E. I. du Pont de Nemours & Co. Inc.

Radiation Effects on Welds and Notches in Plain-Carbon Steels, Stainless Steels and Nonferrous Alloys—D. O. Leeser, Atomic Power Development Associates.

Fast Neutron Effects on Tensile and Hardness Properties of Type 347 Stainless Steel—W. F. Murphy and S. H. Paine, Argonne National Laboratory.

Effects of Radiation Damage on Precipitation Hardening Alloys with Special Reference to Copper-Iron Alloys—A. Boltax, Massachusetts Institute of Technology.

PAINT[32]

Report on Activities of Committee D-1

THURSDAY, SEPTEMBER 20, 2:15 P.M.

Presiding: J. C. Moore (National Director, ASTM), Technical Dir., Coatings Research Group, Bethesda, Md.

W. A. Gloger (Secretary, Committee D-1), National Lead Co., Brooklyn, N. Y.

The D-1 Program on Drying Oils—E. C. Gallagher (National Lead Co.). Vice-Chairman, Subcommittee II.

A Study of the Quality of Water in the Carbon Arc Light and Water Exposure Apparatus—W. F. Singleton (E. I. du Pont de Nemours & Co., Inc.), Subcommittee VII.

Solvents and Thinners—M. B. Chittick (American Mineral Spirits Co.), Chairman, Subcommittee V.

Gloss Measurement—Past, Present and Future—H. K. Hammond (National Bureau of Standards) Secretary, Subcommittee X.

Proposed Methods for the Preparation for Painting of Light Metal Alloy Surfaces—R. I. Wray (Aluminum Company of America) and L. Whitby (Dow Chemical Co.) Subcommittee XXIX.

Progress in Measuring Color Difference—G. W. Ingle (Monsanto Chemical Co), Subcommittee X.

Report on Activities of Subcommittee XII on Latex and Emulsion Paints—M. W. Westgate (National Paint, Varnish & Lacquer Assn.), Secretary, Subcommittee XII.

Progress Review of Subcommittee IX—J. C. Weaver, Sherwin-Williams Co.

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[32] A number of these reports have been published in the *American Paint Journal*.

SYMPOSIUM ON STRUCTURAL SANDWICH CONSTRUCTIONS (*Continued*)[28]

THURSDAY, SEPTEMBER 20, 2:15 P.M.

Presiding: T. P. Pajak (Vice Chairman, Committee C-19, Symposium Chairman), Regional Manager, Hexcel Products Co., Baltimore, Md.
Walter Lovett (Membership Secretary Committee C-19), Sales Engineer, Pittsburgh Corning Corp., Pittsburgh, Pa.

Selection of Materials for Architectural Sandwich Panels—Robert E. Parkinson Kawneer Co.

An Ultrasonic Technique for Non-Destructive Evaluation of Metal-to-Metal Adhesive Bonds—James S. Arnold, Stanford Research Institute.

Recent Developments in Sandwich Construction Including Heat Resistant Materials—R. C. Steele, Hexcel Products, Inc.

Metal-to-Resin Adhesion as Determined by a Stripping Test—W. J. Snodden, Minnesota Mining & Manufacturing Co.

Methods of Testing Sandwich at Elevated Temperatures—E. W. Kuenzi, U. S. Forest Products Laboratory.

SYMPOSIUM ON RADIOACTIVE ISOTOPES[33]

Two Sessions

FRIDAY, SEPTEMBER 21, 9:00 A.M.

(*Sponsored by Committee E-10 on Radioactive Isotopes*)

Presiding: R. B. Stringfield (Past Chairman, So. Calif. Dist.), Consulting Chemical Engineer, Los Angeles, Calif.
C. E. Crompton (Symposium Chairman, Chairman Committee E-10) Assoc. Technical Dir., National Lead Co. of Ohio, Cincinnati

Electroplating and Metal Preservation Studies Utilizing Radioisotopes—Stanley L. Eisler, Rock Island Arsenal Laboratory.

Evaluation of Rubber Deterioration by Means of Radioisotopes—J. F. Young and C. W. Richards, Douglas Aircraft Co., Inc.

The potential of Autoradiography as a Testing Technique—Henry Gomberg, University of Michigan.

Use of Radioactive Tracers in the Study of Soil Removal and Detergency—E. B. Ashcraft, Westinghouse Research Laboratories.

Refinery Scale Applications of Radioactive Tracers—V. P. Guinn, H. R. Lukens, Jr., and D. C.,Wagner, Shell Development Co.

Activation Analysis for Industry—Abel DeHaan, Tracerlab, Inc.

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[33] Issued as separate publication *ASTM STP No. 215.*

SYMPOSIUM ON PAINT

Two Sessions

FRIDAY, SEPTEMBER 21, 9:00 A.M.

Presiding: S. C. Harris, Bradley Paint Co., Los Angeles, Calif.
G. J. Grieve (Secretary, No. Calif. Dist.), Sales Mgr., Transportation Sales, Pacific Paint & Varnish Co., San Francisco, Calif.

Silicones in the Protective Coatings Industry—H. L. Cahn, General Electric Co.[34]

Surface Treatments for Metals Prior to Painting—A. J. Tuckerman, Bradley Paint Co.

Some Aspects of Testing Vinyl Metal Finishes—C. I. Spessard, Bakelite Co., Division of Union Carbide & Carbon Corp.

Protective Coatings for the Railroad Industry—G. J. Grieve, Pacific Paint & Varnish Co.

FATIGUE

Two Sessions

FRIDAY, SEPTEMBER 21, 9:00 A.M.

(Sponsored by Committee E-9 on Fatigue)

Presiding: E. O. Bergman (Vice Chairman, West Coast Committee), Technical Adviser C. F. Braun & Co., Alhambra, Calif.
R. E. Peterson (Chairman, Committee E-9), Mgr., Mechanics Div., Westinghouse Research Labs., Westinghouse Elec. Corp., E. Pittsburgh, Pa.

Effect of a Number of Variables on the Fatigue Properties of High-Strength Steels —G. Sachs, B. B. Muvdi, and E. P. Klier, Syracuse University Research Institute.[15]

Full Scale Wing Fatigue Testing—A. R. Vollmecke, Convair Division, General Dynamics Corp.[35]

Riveted Joints—Fatigue Strength—C. R. Smith and G. D. Lindeneau, Convair Division, General Dynamics Corp.

Determination of Fatigue Crack Initiation and Propagation in a Magnesium Alloy—Robert B. Clapper and J. A. Watz, The Dow Chemical Co.[15]

Axial Stress Fatigue, Creep, and Rupture Properties of Unnotched and Notched Specimens of Heat Resistant Alloys—F. H. Vitovec and B. J. Lazan, University of Minnesota.[15]

SYMPOSIUM ON RADIOACTIVE ISOTOPES[33]

Two Sessions

FRIDAY, SEPTEMBER 21, 2:15 P.M.

Presiding: G. D. Calkins (Honorary Chiarman, Committee E-10), Asst. Division Chief, Battelle Memorial Inst., Columbus, Ohio

Determination of Napthalene in Coal Tar by Dilution Analysis—O. K. Neville, Nuclear Instrument and Chemical Co.

Thickness and Concentration Gaging with Radioactive Isotopes—R. L. Newacheck, J. Kohl, and D. L. Forrest, Tracerlab, Inc.

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[34] To be published in ASTM BULLETIN.

[35] To be published in ASTM BULLETIN.

Application of a Beta Ray Backscatter Thickness Gage to Traffic Paint Wear Studies—Bryant W. Pocock, Michigan State Highway Department.

Will ASTM Standards Be Influenced by Radiation Effects in Metals?—J. C. Wilson and R. G. Berggren, Oak Ridge National Laboratory.[29]

The Problem of Establishing Specifications for Irradiated Organic Materials—Oscar Sisman, Oak Ridge National Laboratory.[29]

Problems of Radiation Dosimetry—V. P. Calkins, General Electric Co., ANP Project.[29]

SYMPOSIUM ON PAINT

FRIDAY, SEPTEMBER 21, 2:15 P.M.

Presiding: L. A. O'Leary (Vice Chairman, Finance Committee), Head, Chemical Engineering & Research, W. P. Fuller & Co., So. San Francisco, Calif.
L. L. Whiteneck (Chairman, Industrial Luncheons Commitee), Vice President and Director, Corrosion Control and Material Research, Plicoflex, Inc.

Evaluation of Paints and Protective Coatings for Municipal Use—J. H. Rigdon, Los Angeles Bureau of Standards.

A Method for the Evaluation of Protective Coatings on Metal Surfaces in Marine Environments—L. L. Whiteneck, Long Beach Harbor Department.

Control of Phosphatizing Systems—Sam Spring, Kelite Corp.

Why Paint Specifications?—Their Tests and Controls—C. F. Pickett, Paint and Chemical Laboratory, Aberdeen Proving Ground.

A New Optical Parameter for Glare-Reducing Window Coatings—R. D. Hitchcock and W. L. Starr, U. S. Naval Civil Engineering Research and Evaluation Laboratory.

FATIGUE

FRIDAY, SEPTEMBER 21, 2:15 P.M.

Presiding: F. M. Howell (Member, Administrative Committee on Papers), Aluminum Research Labs., Aluminum Company of America, New Kensington, Pa.
Paul Kuhn (Chairman, Subcommittee on Aircraft Structural Fatigue Problems, E-9), NACA, Langley Aeronautical Lab., Langley Airforce Base, Va.

Rehibilitation of Fatigue-Weary Structures—J. P. Butler, Boeing Airplane Co.[36]

The Effect of Forming on Mechanical Properties—J. L. Waisman, Douglas Aircraft Co., Inc.[15]

The Effect of Temperature and Frequency on the Fatigue of High Purity Aluminum—J. E. Dorn, University of California.[15]

Uni-Directional Axial Tension Fatigue Tests of Beryllium Copper and Several Precipitation Hardening Corrosion Resistant Steels—M. H. Weisman, J. Melill, and T. Matsuda, North American Aviation, Inc.[15]

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[36] To be published in ASTM BULLETIN.

SUMMARY OF PROCEEDINGS OF BUFFALO SPRING MEETING

During the 1956 Committee Week held in Buffalo, New York, a special technical session was held on Wednesday, February 29, constituting the 1956 Spring Meeting:

Symposium on Properties, Tests, and Performance of Electrodeposited Metallic Coatings

This symposium was sponsored by Committee B-8 on Electrodeposited Metallic Coatings. Two major problems that have faced Committee B-8 are what materials and processes are necessary for a satisfactory electroplate and what is a valid test of a satisfactory electroplate. In an effort to bring up to date the work in this field, the committee sponsored this Symposium on Properties, Tests, and Performance of Electrodeposited Metallic Coatings.

The Society held its Second Pacific Area National Meeting in Los Angeles during the week of February 17–21. At the request of the local committee in charge of the technical program the Symposium of Electrodeposited Metallic Coatings was re-presented during the Pacific Area Meeting, on September 19, 1956, at which time an additional paper by A. L. Alexander on "Evaluation of Phosphate Coatings over Electrodeposited Zinc," covering this further project of Committee B-8, was included. Advantage has been taken of the discussion that took place at both the Buffalo and Los Angeles meetings in the preparation of this publication.

Mr. W. L. Pinner, Houdaille Industries, Inc., Detroit, Mich., acted as Symposium Chairman and presided over the morning session at the Buffalo meeting, and with J. P. Rich, Consultant, Redondo Beach, Calif., at the morning session at the Los Angeles meeting. Mr. R. J. McKay, International Nickel Co. Inc., New York, N. Y., acted as Chairman for the afternoon session at the Buffalo meeting, and Mr. R. B. Saltonstall, the Udylite Corp., Detroit, Mich., acted as Chairman for the afternoon session at the Los Angeles meeting.

The following papers are included in the Symposium:

- Introductory Remarks—W. L. Pinner
- History of ASTM Committee B-8—William Blum
- The Corrosion Behavior and Protective Value of Copper-Nickel-Chromium and Nickel-Chromium Coatings on Steel—C. H. Sample
- Evaluation of Methods Available for Measurement of Surface Luster of Electroplated Coatings—G. B. Bowman
- Recommended Practices for Cleaning Prior to Electroplating—S. Spring
- A Comparison of the Corrosion Behavior and Protective Value of Electrodeposited Zinc and Cadmium Coatings on Steel—C. H. Sample, A. Mendizza, and R. B. Teel
- Evaluation of Phosphate Coating over Electrodeposited Zinc—A. L. Alexander
- Evaluation of Testing Methods for Supplementary Coatings—R. E. Harr
- Atmospheric Exposure of Electroplated Lead Coatings on Steel—A. H. DuRose
- The Standard Salt-Spray Test—Is It a Valid Acceptance Test—A. Mendizza.

These papers with discussion were issued as *ASTM Special Technical Publication No. 197* entitled "Symposium on Properties, Tests, and Performance of Electrodeposited Metallic Coatings."

THE CHALLENGE OF NUCLEAR ENERGY ACCEPTED BY THE ELECTRIC POWER INDUSTRY AND ASTM

ANNUAL ADDRESS BY THE PRESIDENT

C. H. FELLOWS[1]

JUNE 19, 1956

As many ASTM'ers may know, I have spent nearly 35 years working on ASTM's technical committees, the last 25 years as a member of the Society. For nearly 40 years I have been associated with one company in the nation's great electric power industry. Within that time I have seen ASTM Standards—methods of testing and evaluation of materials and specifications—adopted in that industry for its great multiplicity of purposes.

Its construction programs employing cement, concrete, and steel have been controlled by this Society's specifications; its purchases of steel piping used in the generation and transfer of high-temperature steam have been controlled by those specifications; its selection of electrical insulating oils and lubricating oils has come to be made through the application of ASTM-developed test procedures; and the great variety of manufactured equipment comprising components of wood, paper, fabric, plastics, paints, and such metals as lead, copper, aluminum, and silver have been judged as acceptable or have been rejected upon the basis of ASTM Standards.

Mechanical and electrical engineers applied these Standards to the industry's business. Within the period of my association in it, the chemist and metallurgist have come to play ever increasingly important roles. And today a new scientist—the physicist—is called upon to contribute of his fund of knowledge to that of these others in the effort to meet the challenge created by the demand for more and more power.

Perhaps the most important step that has been made in the past 10 years toward the goal of more power, possibly at an even lower cost than prevails at present, has been that concerted effort by industry, and chiefly by the electric power industry, to develop the efficient application of nuclear energy for the generation of electric power.

Until now you have had no clue as to the theme of this address. After all, a theme, as someone once said, is just a point of departure from which a speaker may wander for forty or fifty minutes. I can assure you, I have done just that during the past 18 months while I debated the question of what subject to speak on today. Many of you are more closely associated with the industrial application of nuclear energy than I am, but I believe most of you will be interested in the present status of that effort, begun in 1950, by the Dow Chemical Co. and Detroit Edison Co. to examine the possibilities of the development of an economically competitive nuclear-energy heat source in the generation of electric power.

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[1] Director, Engineering Laboratory and Research Dept., The Detroit Edison Co., Detroit, Mich.

May I take just a few minutes to tell you of this project and, I hope, show you how ASTM with its logical and systematic approach to the materials problems of industry is now diligently concerning itself in this new area of evaluating the measurable physical effects of nuclear radiation of materials.

APDA—Atomic Power Development Associates—is the outgrowth of the original Dow Chemical-Detroit Edison group of associates from which Dow Chemical later withdrew. This group comprises 33 electric power companies and 12 engineering and manufacturing firms. It is a nonprofit membership corporation of United States industries, having as its primary objective the development of the heat of nuclear fission into a commercially practicable means of electric power generation.

Specifically this corporation was commissioned to design and develop a liquid-metal-cooled, fast breeder reactor having a gross electric output of 100,000 kw with steam conditions defined as 600 psi and 750 F. The estimated cost was to be $45,000,000.

PRDC—Power Reactor Development Company—comprising 18 electric power companies and 8 engineering and manufacturing companies was organized to build, own, and operate the reactor designed by APDA.

APDA's objective is a continuing one. While PRDC builds and operates this initial reactor, the earlier group will continue its studies, because it is recognized that this first power reactor will not be an economically competitive unit and that through experience with it, new designs will be born that will enable the engineers to more closely approach the ultimate objective.

Even today there will be some who question the need for developing nuclear power. Many have wondered why such large sums of money and so many man-hours have been expended in its development. Actually, there are three very important reasons.

First, our ever-increasing requirements for energy dictate that we supplement, and use more effectively, our present conventional fuels. During the past ten years, the electric power industry has more than doubled its output and has increased its conventional fuel consumption nearly 100 per cent. It is expected that an equal growth will result during the next decade. With this increase in fuel consumption, we must concern ourselves with future availability of conventional fuels. Data compiled by the Atomic Energy Commission indicate that the known world deposits of uranium and thorium represent, assuming full utilization, some 23 times the heat equivalent of the world's conventional fuel deposits. This then constitutes an urgent reason for the development of atomic power.

The second reason for developing this type of energy is the possibility of producing power at a lower cost. We believe that there lies within the atom, this potential.

Power costs vary greatly, even throughout the United States, depending largely upon the availability of fuel. Therefore, what is economic power in one locality may not prove to be economical in other areas or other countries. It is evident that in the New England area, where fuel costs are as high as 40 to 50 cents per million Btu, nuclear power can be justified much more readily than in the Southwest, where fuel costs are on the order of 10 to 15 cents per million Btu.

Then too, coal costs are ever increasing, and consequently, if power companies are to maintain low rates, as they have in the past, it is imperative that further engineering techniques be developed. We look to nuclear energy as a means of eventually producing lower-cost power, provided, of course, that costs of labor, taxes, and construction materials remain at or near present levels.

The third reason for developing nuclear energy is the political aspects of the world race for the peaceful use of the atom. This is probably one of the strongest motivations for our Government to solve this problem. President Eisenhower's international program and the formation of the Fund for Peaceful Atomic Development are significant steps toward this end.

The APDA group in Detroit is now working on the development of what is called a breeder-type reactor in which more fissionable fuel is created than is burned. Actually very little practical experience is available with the breeder-type reactor. Argonne National Laboratories constructed and has operated since late in 1951 the EBR I (Experimental Breeder Reactor No. 1) at the National Reactor Testing Station, Arco, Idaho. They are now designing a second breeder reactor of 60 Mw heat output to be known as EBR II.

Fuel costs are greatly reduced with this type of operation, but unfortunately, there are offsetting disadvantages. Fixed charges are high, associated with a large inventory of valuable materials. Expensive fuel processing also is a major problem with this type of reactor.

Each type of reactor has its own characteristic advantages as well as disadvantages. At this period in reactor technology, it is not possible to state which of these represents the best over-all solution to economical nuclear power. Because of the many unknowns, it was necessary that additional experience be obtained with a large variety of reactors. During the early part of 1954, the AEC announced its 5-yr reactor program. This program encompassed the development of five different reactor concepts. It was undertaken in an effort to assist in advancing reactor technology and civilian use of nuclear power.

This program includes:

1. pressurized water reactor,
2. experimental boiling water reactor,
3. sodium-graphite reactor experiment,
4. experimental breeder reactor No. 2,
5. homogeneous reactor.

Paralleling this AEC reactor program and in addition to it, there is the so-called "Reactor Demonstration Program for Industry." This is being carried on by private industry and includes numerous utilities, equipment manufacturers, engineering and construction firms, chemical companies, shipbuilding companies and others working together to advance the field of reactor technology and nuclear energy. In this program, commercial reactors of each of the types mentioned earlier are planned for construction.

In the case of the *pressurized water reactor*, the Yankee Atomic Electric Co. including 12 utility companies is considering constructing a plant at Rowe, Mass., that will have a capacity of about 130,000 kw.

The Consolidated Edison Co. of New York is also considering constructing a plant using a reactor of this type at Indian Point, N. Y. This plant will have a capacity of 150,000 kw.

The Commonwealth Edison Co. and its associates are planning construction of a plant at Dresdon, Ill., using the *experimental boiling water reactor* that will have a capacity of 180,000 kw.

The *sodium-graphite reactor experiment* is a responsibility of the North American Aviation Co. It will be constructed at Santa Susana, Calif. The Southern California Edison Co. will install turbine equipment to utilize the steam.

The Consumers Public Power District of Nebraska has AEC approval to install a plant of this type that will have a capacity of 75,000 kw.

The Nuclear Power Development Co. including The Detroit Edison Co. and 25 associates will construct the *developmental breeder reactor* plant south of Detroit. It will have a capacity of 100,000 kw.

Besides the two *homogeneous reactors* being developed and studied by the Oak Ridge National Laboratories there is one under consideration jointly by the Pennsylvania Power and Light Co. and Westinghouse for large scale power generation. Reactors of this type are significantly different from the others in that the finely divided fuel, either uranium or thorium, is suspended in water and pumped through the reactor vessel.

This is a program in which over $300,000,000 of private money is being spent. It is the largest research effort ever undertaken in which the electric power industry is taking the leading role.

As I stated earlier, APDA designed and developed a liquid-metal-cooled, fast breeder reactor; PRDC will build and operate it. It is to be built on a site some 30 miles south of Detroit near the west shore of Lake Erie. Ground is expected to be broken for this new privately owned plant on August 8, 1956, the anniversary of the Geneva Atomic Energy Conference in 1955. This anniversary date, we believe, will be historically significant. It will be completed in 1960.

ASTM is also fully aware of the impact which the extensive use of nuclear energy will make in regard to materials testing programs. It may interest you to know that ASTM has already undertaken talks with AEC representatives for the purpose of exploring ways by which the Society with its organized committee setup might best serve industry and AEC in the area of developing standard tests for the structural materials of atomic reactors and materials of accessory equipment.

It was the consensus of a meeting attended by representatives of selected ASTM Technical Committees that the Society proceed at once to initiate programs within the framework of its committee organization toward the end that standard test methods and ultimately specifications could be developed. This recommendation was approved by the Board of Directors at its January meeting. An Advisory Committee was appointed. It will function as an administrator and coordinator that will guide ASTM's activities in this new field, both with respect to power and other applications. Our membership can feel assured that ASTM is taking the significant part it should take in the development of this important new source of power for American as well as worldwide industry.

Although we have made significant steps forward in reactor technology, we still have a long way to go. The nuclear power industry stands today where conventional-fueled steam power did fifty years ago.

Great advances have taken place in the production of electrical energy largely because of the efforts of the electric power industry and the manufacturers of electrical apparatus and equipment. I am confident that equally important developments in power generation using nuclear reactors as heat sources will also come in the years ahead.

I am equally confident that, as our ASTM Technical Committees contributed significantly to the development of materials for present-day power plants, those same committees augmented in some instances by new personnel will make a like contribution in this area of generation using nuclear energy.

Throughout this entire discussion I have emphasized the striking progress the electrical power industry has made in the direction of developing and adapting to its own purposes this energy from the atom.

In closing, I should like to leave this thought with you: I quote:

> Man is not master of the universe because he can split the atom. He has split the atom because he believed in his own unique mastery. Faith led to the material achievement, not the achievement to the faith.

ANNUAL REPORT OF THE BOARD OF DIRECTORS

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Someone has said, "A business succeeds not because it is big, or because it has been long established, but because there are men and women in it who live it, sleep it, dream it, and build great future plans for it." Basically, this is the underlying reason for the past, present, and continuing successful work and expansion of our Society. Figuratively and actually there are many of our officers

HIGH LIGHTS, 1955 FINANCIAL OPERATIONS

	1955	1954	1953	1952
Gross Receipts:				
Membership Dues	$252 100	$236 400	$233 700	$226 300
Sales of Publications	521 500	470 500	512 300	338 500
Other	78 200	87 700	68 600	83 600
Total	851 800	794 600	814 600	648 400
Expenditures:	$305 400	$276 900	$237 800	$211 800
Salaries	350 000	337 500	323 800	259 800
Printing	196 000	196 600	159 500	148 400
Other				
Total	851 400	811 000	721 100	620 000
Favorable Operating Balance	$400	...	$93 500	$28 400
Operating Deficit	...	$16 400	...	...
Net Assets:				
Real Estate	$277 700	$295 200	$244 800	$184 800
Investments and Cash	880 500	797 000	776 950	733 900
Equipment	29 500	29 700	22 800	22 500
Total	1 187 700	1 121 900	1 044 550	941 200
Asset Equity of Each Member	$147	$145	$138	$128
Number of Members	8 079	7 762	7 581	7 342

	1955	1954	1953	1952
Pages of Publications	18 742	8 088	8 518	15 747
Number of Employees	69	65	60	56

and members who devote a considerable portion of their time, much of it outside office hours, to the welfare of ASTM. No other technical or professional organization has such a high percentage of its members and workers actually doing things on committees, or preparing papers, or performing related functions, as does ASTM.

Including the upwards of 5000 members of our committees who themselves are not actual members or official representatives of company members, it is estimated that 70 per cent of the 13,500 men in the Society are serving on technical or other committees. With these committees occupying such an essential place in the Society, it is a corollary that we progress because of the consistent work they are doing.

What new activities were initiated in the Society in recent months as a result of the efforts and enthusiasm of our members? Very briefly, in the following portions of this report, are noted some of these developments. Several new committees were organized or authorized as noted under Technical Activities. The tremendous volume of published material, over 18,000 pages, bespeaks well the results of research and standardization work.

Our membership, reaching a new high, includes 775 new members who joined in 1955—by far the largest ever. The preceding table showing highlights of financial operations indicates a steady growth in net assets and notes that 1955 was the year of our highest income.

As a Society for materials, and not solely concerned with testing, it is an obligation of ASTM that it welcome representation from other organizations either concerned with or doing work relating to materials. Closer relations with a number of important professional and trade groups have been established in 1955. We see an increasing interest in our technical committee work by industry in the western states and gradually, with distance being broken down by more ready means of communication, more of the West's leading engineers are becoming active in our work.

All in all, 1955 was a satisfactory year. But standards are not static. Research, too, has to be aggressive and the pressing demands for new or improved materials continue. It may be that the most basic factor responsible for the progress made is a realization that our work will never be done; that always there will be better ways of testing and evaluating materials, that continually we can improve their quality; and finally that despite a mild sense of satisfaction of where the ASTM's 58 years have brought us, much more remains to be done and with the tempo of demand rising, we must be able to do our work faster and more efficiently.

Technical Activities

The past year was marked with a number of important new technical activities. As noted below, new main committees were formed and in addition to these some of the older committees expanded their work to cover important new segments of the materials fields with which they are concerned. An earnest attempt is made through the annual reports of the committees and by news accounts in the ASTM BULLETIN to keep the members informed. Some of the important new developments are noted here.

New Activities

Hydraulic Fluids:

To undertake needed research and standardization work covering both synthetic and petroleum type hydraulic fluids, a new Technical Committee N has been organized in Committee D-2 on Petroleum Products and Lubricants. This work will encompass test methods, specifications, and nomenclature and also may involve research work leading to a better knowledge of the properties of these materials which are steadily growing in importance. The committee includes representatives not only of leading producers and consumers but also of other organizations concerned.

Putty, Glazing, and Caulking Compounds:

Culminating considerable discussion on coverage of glazing and caulking compounds, a new subcommittee was organized in Committee D-1 on Paint, Varnish, Lacquer, and Related Products. This group, which will be concerned with putty and glazing and caulking compounds, has indicated that its long-range program would involve the following: (1) glazing compounds for metal sash and wood sash, (2) caulking compounds, (3) putty for metal and wood sash, and (4) extruded-type caulking compounds. Reference to this work appears on page 37 of the April ASTM BULLETIN.

Carbon Black:

The April BULLETIN, page 45, describes steps leading to the new Committee D-24 on Carbon Black, to be formally organized during the Annual Meeting. The scope of the committee, as approved by the Board of Directors, is as follows:

Scope.—The promotion of knowledge of the properties of those materials generally known as carbon blacks, including stimulation of research and the development of methods of test, classification, specifications and nomenclature. These activities will be coordinated with those of relevant committees of ASTM.

Carbon black is used in a variety of industries and the new committee will have the benefit of important recent work in the industry on test methods. It is expected these proposed methods will be published as information in the ASTM BULLETIN to elicit comments.

Sorbent Mineral Materials:

Consideration of this field started with a suggestion from those concerned with absorbent floor cleaning materials. As a result of study and conferences a new technical committee has been authorized with the following scope:

Scope.—The definition of terms and the formulation of methods of test covering sorptive mineral materials as used for surface maintenance and safety and the stimulation of research to accomplish the foregoing purposes.

While the original proposal relates specifically to the clay type of material (fullers earth) the scope as outlined envisions diatomaceous earth and others as well.

The Directors also approved the proposed personnel of the committee, which will be announced.

Casein:

During the year the Board of Directors took favorable action with respect to the organization of a new committee on casein which would develop definitions of terms and formulate test methods and specifications for industrial casein. The establishment of this committee is being implemented through the customary procedure of the appointment of a steering committee to bring in specific recommendations on personnel. While the work will initially be confined to casein, it is possible it will later be expanded to include other proteins.

Magnet Wire:

The interest in magnet wire has increased considerably during the past few years, particularly in view of the introduction of a number of newer types of insulation. The Society has been represented on the Sectional Committee on Magnet Wire (C9), functioning under the sponsorship of the National Electrical Manufacturers Association. In addition to this, a new subcommittee has been established in ASTM Committee D-9 on Electrical Insulating Materials to deal with the methods of testing magnet wire, and the representation of the Society on the sectional committee has been increased in order to tie in this new work effectively with the work of the sectional committee. It is not proposed to carry out work on specifications for magnet wire independent of the sectional committee. Specifications for the conductor for use in magnet wire are covered by ASTM Committee B-1.

Materials Used in Nuclear Energy Work:

To guide and counsel the Board of Directors and the technical committees on the involved problems which will develop as research and standardization studies get under way on materials used in nuclear energy work, both in reactors and in other areas, an extensive conference was held in New York City on November 11, 1955. Numerous technical committees and other organizations were represented. It was the consensus that work on materials should be undertaken promptly and that it was quite logical that these activities generally be handled in the respective interested technical committees. In view of the importance of the subject, a special administrative committee was set up, the personnel of which is given below. Following considerations in this group and participation in a general conference called by the American Standards Association, a letter was sent to the officers of ASTM technical committees with the request that they advise of any proposed projects in this field.

Committee B-2 is considering the possibility of writing specifications for zirconium and beryllium and nuclear fuel elements such as uranium; Committees A-1 and A-10 have included some steel compositions in certain of the austenitic steel specifications for extra low carbon grades and also others where the tantalum content is limited; Committee A-10 has appointed a special committee to deal with the materials in its particular field; Committee D-19 is determining the concentration of some of the long-life isotopes in industrial waste water; a paper was presented at the recent meeting of Committee C-8 on some of the problems associated with refractories; Committee C-21 on Ceramic Whitewares has organized a section in this field under their Research Subcommittee; Committee D-20 on Plastics has established a group to study the effect of radiation on plastics. Committee E-7 on Non-Destructive Testing is very much interested in a proposed Symposium on Non-Destructive Testing in the Nuclear Energy Field; and, of course, Committee E-10 on Radioactive Isotopes is interested as indicated by the revised title and scope set forth below as approved by the Board.

Proposed Title: Committee E-10 on Radioisotopes and Radiation Effects.

Proposed Scope: To promote the knowledge of the use of radioisotopes in materials testing and the investigation of the changes in the properties and constitution of materials as a function of exposure to radiation by:

1. Aiding and advising the technical committees of the Society in the preparation of methods and development of instruments of testing.

2. Preparing methods of testing not coming within the scope of other technical committees.
3. Standardizing the nomenclature and definitions used in or relating to testing methods or instruments in cooperation with other committees.
4. Performing such other functions as may advance, improve, standardize, and unify the procedures of materials testing in the fields of radioisotopes and radiation effects.

From the above, it will be seen that Committee E-10 is prepared to advise the other committees and help in areas where the existing technical groups may need such assistance, but it is expected that the current committees will handle their full responsibilities as they have done when other problems concerning their field have arisen.

The personnel of the Special Administrative Committee on Nuclear Problems is as follows:

N. L. Mochel (chairman), Westinghouse Electric Corp.
G. D. Calkins, Battelle Memorial Inst.
C. L. Clarke, Timken Roller Bearing Co.
M. A. Cordovi, Babcock & Wilcox Co.
G. M. Kline, National Bureau of Standards
F. L. LaQue, International Nickel Co.
F. C. Linn, General Electric Co.
J. C. Robinson, Atomic Energy Commission (now General Electric Co.)
C. C. Woolsey, North American Aviation Co.
J. R. Townsend, Sandia Corp.
H. A. Wagner, Detroit Edison Co.

In activities of this kind it is always important to have available in useful form the latest information and it is of direct interest that an extensive symposium with at least two sessions will be held during the Society's second Pacific Area National Meeting, on September 20. Under the joint auspices of Committee E-10 and the Materials Committee of the Atomic Industrial Forum, a dozen papers will be presented summarizing and providing best information on radiation effects on materials. At the same time there will be another extensive symposium on radioactive isotopes.

Microchemical Apparatus:

A new Subcommittee on Microchemical Apparatus has been organized to function as a part of Committee E-1 on Methods of Testing to carry on the standardization activities formerly handled in a Division of the American Chemical Society. The transfer of this activity to the ASTM is the result of action taken by the ACS to discontinue standardization activities, except on the subject of reagents, and to transfer such projects to groups organized for this purpose such as the ASTM.

The personnel of this group and other information on its activities were given in the February issue of the ASTM BULLETIN.

This apparatus is very specialized in design and in size and for this reason there is little likelihood of overlap with other E-1 work dealing with apparatus, but in any case there is suitable cross representation in membership.

The Society is pleased to sponsor the standards work of this important group which is vital to many branches of industry. Meanwhile the corresponding ACS committee will continue its other activities.

Under Consideration

Independent discussions under way or in the technical committees, some of which have been noted by the Board of Directors, eventually may lead to additional formal standardization or research projects. Notes on a few of these follow:

Manganese Dioxide:

A request from a group working on test methods and requirements for manganese dioxide of battery grade that ASTM set up a new committee, is being explored.

Contacts are being made with another association interested in this activity.

Cermets:

Several technical committees are concerned with the materials known as cermets. Committee C-21 on Ceramic Whitewares has been considering a definition and Committee B-9 on Metal Powders and Metal Powder Products is also following this subject. Committee C-22 on Porcelain Enamel and the Joint Committee on Effect of Temperature on the Properties of Metals have a vital interest. A conference may be called to recommend specific responsibility for these materials. The Board of Directors is following this work closely, being cognizant that ASTM should take a leading role in its development.

Flexible Barriers:

Growing interest in flexible barriers and packaging materials led to a review of this proposed activity in ASTM Committee D-10 on Shipping Containers. Meanwhile the National Flexible Packaging Assn. has indicated interest in having ASTM become active in this field. Further discussions are to be held with Committee D-10 which has one subcommittee concerned with interior packing.

Committee Titles, Scopes, and Jurisdiction

Two technical committees have had their titles changed, as follows:

The title of Committee C-21 will now read "Ceramic Whitewares and Related Products." A corresponding change is being made in the Scope of the Committee.

Committee E-10 on Radioactive Isotopes will now operate under the title "Radioisotopes and Radiation Effects." Reference is made to the work of this committee in a preceding paragraph discussing materials used in nuclear energy work.

The following committees have prepared revised scopes which have been approved by the Board:

Committee D-9 on Electrical Insulating Materials:

Considering developments in the field of electrical insulating materials it has seemed wise to revise the scope of Committee D-9 from its present form, namely,

> Development of methods of sampling and testing, and formulation of specifications for electrical insulating materials, including research concerning the properties of such materials and products,

to read as follows:

> *Scope.*—(1) Development of methods of sampling and testing, and formulation of specifications for electrical insulating materials, comprising materials designed as electrical insulation and those grades of other dielectric materials designed with controlled dielectric, piezoelectric, or magnetostrictive properties;
>
> (2) Research, comprising studies of fundamental properties and application problems, on electrical insulating materials.

It had been proposed that responsibility for review of tests and specifications for dielectric materials as prepared by other committees would be stated, but because any technical committee of the Society may review specifications and tests in which it has an interest, this particular statement was considered unnecessary and was not included since it might have been construed as implying that Committee D-9 was assigned full jurisdiction.

Committee D-11 on Rubber and Rubber-Like Materials:

Because of its interest in reference standards and compounding ingredients, Committee D-11 requested that its scope be expanded to read as follows by the addition of the words in italics:

> *Scope.*—Formulation of specifications, methods of test and definitions of terms pertaining to rubber and rubber-like materials, *the compounding ingredients as used therein* and *the* products therefrom, including research concerning the properties of such materials and products.

This scope was reviewed by those concerned with the new Committee on Carbon Black referred to earlier and it is felt that there is no conflict of interest.

Concrete Drain Tile

Problems in handling standards requirements for concrete drain tile, together with strong recommendations from those concerned with these materials led to reassignment of responsibility to Committee C-13 on Concrete Pipe. Formerly clay and concrete drain tile were handled in Committee C-15 on Manufactured Masonry Units, which had responsibility for the single Standard Specification C-4 covering both materials. Clay materials will continue to be the responsibility of Committee C-15. The two committees have been asked by the Board to recommend the best means of coordination.

District and Related Activities

Thirty local ASTM meetings were held during the past year, including 24 district meetings and 6 meetings held in areas not currently incorporated in any formal district. New areas covered included Salt Lake City, Vancouver, and New Orleans. President C. H. Fellows presented his district address "Research in the Electric Power Industry (Generation—Transmission—Distribution)" fifteen times. Although a number of meetings were sponsored solely by ASTM groups, the majority were cosponsored with other societies such as AIEE, ASME, ASM, ACS, AIChE and the Western Society of Engineers.

Dr. Fritz V. Lenel, 1955 Gillett Memorial Lecturer, presented an abridged version of his lecture on "Powder Metallurgy—Now" in Chicago, St. Louis, Los Angeles, San Francisco, and Dallas.

Several of the districts have taken interesting approaches to the problem of developing technical programs. The New England District has for the past several years held its Spring Meeting at one of the colleges or universities in the area, usually preceded by a tour of the engineering facilities in the afternoon. The New York District is trying the idea of having a particular company sponsor a program such as the January 11 meeting "A Night With Bell Telephone Laboratories."

The Philadelphia District acted as the host for the 1955 Annual Meeting, sponsoring the ladies entertainment and underwriting the entertainment which followed the annual dinner. This same group has also agreed to take on a similar responsibility for this year's meeting.

In addition to sponsoring technical sessions, arranging for Annual Meeting and Spring Meeting dinners and entertainment, various districts have been active in sponsoring student membership prize awards, participating in such activities as science fairs and local engineering councils, and cooperating with the Society's Advisory Committee on Corrosion.

The Administrative Committee on District Activities held a meeting during the 1955 Annual Meeting and sponsored a breakfast meeting to which district council officers were invited. These meetings have been helpful, serving as an excellent means for interchange of information and ideas for promotion of the Society and its activities.

The accompanying table does not list the details of the local meetings, all of which were described in the ASTM BULLETIN but it does give the locations and meeting dates.

There is considerable interest in the establishment of a separate district in

Annual Meeting:		
1955 Annual Meeting	June 26–July 1, 1955	Atlantic City, N. J.
Committee Week and Spring Meeting:		
1956 Committee Week and Spring Meeting	February 27–March 2, 1956	Buffalo, N. Y.
District or Special Local Meetings:		
Chicago District	October 12, 1955	Chicago, Ill.
St. Louis District (jointly with Engrs. Club and ASM)	October 13, 1955	St. Louis, Mo.
New England District	October 27, 1955	Cambridge, Mass.
Southern California District (jointly with ASM)	October 27, 1955	Los Angeles, Calif.
Pittsburgh District	October 27, 1955	Pittsburgh, Pa.
Northern California District (jointly with ASME)	October 28, 1955	San Francisco, Calif.
Southwest District (jointly with ASM)	November 3, 1955	Dallas, Tex.
Ohio Valley District (jointly with AIEE)	November 17, 1955	Cincinnati, Ohio
Philadelphia District (jointly with ASQC)	November 18, 1955	Philadelphia, Pa.
New York District (jointly with AIEE)	January 11, 1956	New York, N. Y.
St. Louis District (jointly with Mo. Society of Prof. Engrs)	January 24, 1956	St. Louis, Mo.
Washington, D. C. District (jointly with Baltimore Engrs. Club)	January 27, 1956	Baltimore, Md.
Philadelphia District	February 10, 1956	Philadelphia, Pa.
Western New York-Ontario District	February 28, 1956	Buffalo, N. Y.
Southwest District	March 1, 1956	New Orleans, La.
Southwest District (jointly with AIEE branch groups)	March 5, 1956	Houston, Tex.
Southern California District (jointly with ASME)	March 8, 1956	Los Angeles, Calif.
Nothern California District (jointly with AIEE)	March 13, 1956	San Francisco, Calif.
Salt Lake City, Utah (jointly with ASME)	March 19, 1956	Salt Lake City, Utah
Richland, Wash. (jointly with ASM, ACS, AIChE, ISA, AIEE)	March 22, 1956	Richland, Wash.
Spokane, Wash. (jointly with ACS, AIEE, ASME, ASCE, AIME, Washington Society of Professional Engineers, Spokane Technical Professional Council)	March 23, 1956	Spokane, Wash.
Portland, Wash. (jointly with ASM)	March 27, 1956	Portland, Wash.
Vancouver, B. C. (jointly with CIC, NACE)	March 29, 1956	Vancouver, B. C.
Chicago District	April 11, 1956	Chicago, Ill.
Cleveland District	April 12, 1956	Cleveland, Ohio
New York District	April 17, 1956	New York, N. Y.
Detroit District	April 25, 1956	Detroit, Mich.
Pittsburgh District	April 26, 1956	Pittsburgh, Pa.
New England District (with Univ. of Mass.)	May 4, 1956	Amherst, Mass.

the southeastern part of the United States, and undoubtedly efforts will be made to accomplish this end in the next year or two.

National Meetings

1955 Annual Meeting

A figure which is ever astounding is the number of committee meetings held during an annual meeting of the Society. During the 58th Annual Meeting in Atlantic City, June 26–July 1, at Chalfonte-Haddon Hall, there were over 700 meetings of the Society's committees, frequently sixty of these running simultaneously. Attendance at this Annual Meeting was 2537, not including 234 ladies. It was necessary to hold 32 technical sessions for the presentation of the 93 papers and 66 reports. Features of the meeting were covered in the July, 1955 ASTM BULLETIN. The Philadelphia District again acted as gracious hosts, providing a fine program of entertainment for the ladies and sponsoring the annual dinner. This group is again handling these features at the 1956 meeting.

Since this was the last meeting prior to the issuance of the triennial Book of Standards, there were a large number of actions on standards and tentatives as detailed in the committee reports and summarized in the BULLETIN and in the report of the Administrative Committee on Standards.

1956 Committee Week and Spring Meeting

The 1956 Committee Week was held somewhat later than usual to avoid as much winter weather as possible in the Western New York area. Buffalo was the city and the meetings from February 27–March 2 were very successful. There were some 350 meetings of committees and a notable Symposium on Electrodeposited Metallic Coatings (sponsored by Committee B-8). The Western New York-Ontario District sponsored a social hour and dinner in honor of the President on Tuesday, February 28. There was an interesting talk on Sports Car Racing with motion pictures. Total registered attendance for the week was 1294.

Second Pacific Area National Meeting

What promises to be the Society's most extensive technical meeting is scheduled for Los Angeles throughout the week beginning September 16. This is the second national West Coast meeting, the first was held in October, 1949, in San Francisco.

Some 250 technical papers comprising about 45 technical sessions will be the main feature. Subjects of intense current concern in the field of materials will be covered. Full details are being furnished all members and committee members and others interested in a special brochure to be mailed early in June.

Seven special Industry Luncheons and a President's National Luncheon have been scheduled. There is a most interesting entertainment program for the ladies. All sessions and luncheons will be at the Hotel Statler, well equipped for a meeting of this kind.

That a meeting of this scope should develop indicates, first of all, the intense concern with growing problems in the field of materials, radioactive isotopes, materials for nuclear energy applications, non-destructive testing, and many others; but it is a reflection, too, of the enthusiasm with which our technical committees planned and arranged many of the symposiums and sessions. However, the extent of the meeting is due particularly to the intense interest and the hard work and enthusiasm of the many West Coast members, especially in the Los Angeles area, who look upon this meeting somewhat as a challenge to insure that the Society and its work is brought forcibly to the attention of the West. Further details of the meeting are being given in the ASTM BULLETIN, but the advance brochure will portray the great scope of the features.

The papers for the meeting are not being preprinted but are being duplicated in quantity for distribution to those who register at the meeting. It is expected that papers in most of the sessions and symposiums will be published in special pamphlet form following the meeting. The members will be kept fully informed concerning these plans.

Future Meetings

Annual Meetings of the Society have been scheduled as follows: June 17–22, 1956; June 16–21, 1957—Atlantic City, N. J.; June 22–27, 1958—Boston, Mass.; June 21–26, 1959, June 26–July 1, 1960—Atlantic City, N. J.

Committee Week and Spring Meetings: February 4–8, 1957, Philadelphia, Pa.; February 9–14, 1958, St. Louis, Mo.; February 2–7, 1959, Pittsburgh, Pa.; February 1–5, 1960, Chicago, Ill.; January 29–February 3, 1961, Cincinnati, Ohio.

Membership

On May 1, 1956, the membership of the Society totaled 8323, compared with a total of 7911 one year ago. Gains and losses in various classes of membership are shown in the following table:

Class of Membership	Membership		Losses				Additions		Total		
	May 1, 1955	May 1, 1956	Resignations	Dropped	Death	Transfer	Transfer	Election	Loss	Gain	Increase
Honorary	21	22	...	...	3	...	4	...	3	4	1
Perpetuity and Life	11	13	...	...	...	...	2	...	...	2	2
Sustaining	345	375	2	1	...	1	27	7	4	34	30
Company, Firm, etc.	1797	1859	38	16	...	29	9	136	83	145	62
Individual, etc.	5652	5919	233	98	51	14	7	656	396	663	267
Associate (formerly Junior)	85	135	3	12	...	5	...	70	20	70	50
Total	7911	8323	276	127	54	49	49	869	506	918	412
Student	454	530	22	208	1	27		334	258	334	76

It is interesting to compare our present membership of 8323 with the figures for ten and twenty years ago; in 1946 our total membership was 5993, and in 1936 it was 3741.

Concerning the calendar year 1955, the number of new members was higher than any previous year, totaling 775. The previous nine years ranged from 602 to 686. However, losses were considerably higher due to more deaths, higher resignations, and more members dropped for nonpayment of dues. The net gain of 317, however, was higher than in any previous year except 1945 and 1946, the best years.

The year was marked, too, by large increase in the number of sustaining members which rose from 275 on January 1, 1955 to 359 on December 31.

Associate Membership:

It is too early to assess the significance of a major change in our structure during the year, namely, dropping "Junior" membership and creating the new "Associate" class which extends to 30 years of age. Considerable promotional work has been done, and will continue, to make this more widely known, particularly in the engineering faculties. We are having good cooperation from professors in many of the leading schools. One of our objectives here was to attract younger engineering faculty members.

Sustaining Members:

There have been 33 new sustaining members in the period from May, 1955 to May, 1956, two resignations, and one sustaining member transferred to company class. The new sustaining members are:

Ansul Chemical Co.
Atlantic Coast Line Railroad Co.
Automatic Electric Co.
Bird and Son, Inc.
Cameron Iron Works, Inc.
Canadian Westinghouse Co., Ltd.
Cities Service Petroleum, Inc.
Climax Molybdenum Co.
Cluett, Peabody and Co., Inc.
Coors Porcelain Co.
DeLaval Steam Turbine Co.
Driver, Wilbur B., Co.
Erie Forge and Steel Corp.
Fairbanks Co., The
Fels and Co.
Ferguson Co., H. K.
Freed Transformer Co.
Harbison-Walker Refractories Co.
Interlectric Corp.
Kollsman Instrument Corp.
Longhorn Portland Cement Co.
McDonnell Aircraft Corp.
Metal and Thermit Corp.
Mycalex Corporation of America
National Portland Cement Co.
Ohio-Apex, Division of Food Machinery and Chemical Corp.
Ohio Edison Co.
Peerless Cement Corp.
Pennsylvania Forge Corp.
Pennsylvania Power and Light Co.
Plastic Metals Division, National-U. S. Radiator Corp.
St. Louis-San Francisco Railway Co.
Stromberg-Carlson Co.

50-Year Members:

There are two individuals and eleven organizations as noted below that have been connected with the Society continuously for 50 years, having joined in 1906:

Charles E. Fuller
Earl B. Smith

American Bureau of Shipping
American Steel Foundries
The Anaconda Co.
Cornell University Library
The Iron Age
Norfolk & Western Railway Co.
Pittsburgh Plate Glass Co., Paint Division
Southern Pacific Co.
United States Testing Co., Inc.
University of Kansas Library
University of Melbourne Library

These members will be recognized at the Annual Meeting.

40-Year Members:

There are 38 members who this year have completed 40 years of membership, bringing the total number of certificates issued to the "Forty-Year Club" to 352. The members who will receive the 40-Year Certificates at this Annual Meeting are as follows:

American Chain and Cable Co., Inc.
American Concrete Pipe Assn.
The American Institute of Architects, Cincinnati Chapter
American Manganese Bronze Co.
The American Metal Co., Ltd.
American Pipe and Construction Co.
Arthur O. Austin
Boston & Maine Railroad
Sumner E. Campbell
Canadian General Electric Co., Ltd.
The Consolidated Mining and Smelting Co of Canada, Ltd.
Continental Foundry and Machine Co.
The Detroit Edison Co.
Duquesne Slag Products Co.
Lewis A. Eisenhard
The Firestone Tire and Rubber Co.
Arthur Percy Morris Fleming
Roy M. Green
Herman A. Holz
Hudson Wire Co.
International Smelting and Refining Co., Raritan Copper Works

Frank H. Jackson
The Kerite Co.
Lloyd McEntire
Narragansett Electric Co.
The Ohio Brass Co., Barberton Division
Ontario Department of Highways
Pennsylvania Forge Co.
City of Philadelphia, Department of Wharves, Docks and Ferries
Portland Cement Assn.
City of Portland, Department of Public Works
John McE. Sanderson
Socony Mobil Oil Co., Inc.
Jerome Strauss
Texas Agricultural and Mechanical College, Civil Engineering Dept.
United States Steel Supply Division, United States Steel Corp.
Wylie B. Wendt

Deaths:

The Society has lost the following 54 members through death (figures in parenthesis are dates of membership):

Amberg, Charles R. (1944)
Anson, I. Arthur (1948)
Bagsar, Aaron B. (1923)
Bass, Frank L. (1949)
Bateman, Frederick W. (1903)
Cochenour, George E., Jr. (1943)
Condron, Theodore L. (1900)
Cowell, Paul John (1948)
Crawford, C. H. (1914)
Cummings, A. E. (1936)
*Curtis, Harvey L. (1923)
Dixon, Enslo Smith (1925)
Dow, Allan W. (1898)
Duchacek, Charles F. (1941)
Dunwoody, G. C. (1953)
Egloff, Gustav (1926)
Fahy, Frank P. (1917)
Flood, Walter H. (1915)
French, Herbert J. (1938)
*Harvey, Dean (1913)
Hollinger, E. R. (1948)
Jones, John Clifford (1937)
Jones, Valens (1948)
*Lester, H. H. (1936)
Libbey, Valentine B. (1937)
Loos, C. E. (1940)
Lynam, Charles (1927)
Mapes, Fred S. (1951)
Meyers, A. M. (1938)
Miessner, Harmon S. (1939)
Mooney, Thomas J. (1948)
Morris, Arthur D. (1950)
Naegely, William H. (1927)
Nichols, M. F. (1939)
Noyes, R. A. (1952)
Pafenbach, A. Roy (1946)
Payne, Frank E. (1944)
Powers, Henry R. (1916)
Reed, Harry S. (1940)
Rogers, P. C. (1936)
Sheard, Harry J. (1946)
Shilstone, Herbert M. (1922)
Smith, Don W. (1948)
Smith, Frank C. (1947)
Styri, Haakon (1920)
Swain, Stephen M. (1940)
Sweeney, Edmond L. (1946)
Von Hambach, E. (1923)
Walsh, L. C. (1944)
Webeck, Russell C. (1946)
Weiller, P. G. (1945)
Widemann, R. V. (1948)
Witsberger, Clarence J. (1948)
Wood, Alan, 3d (1908)

In addition to the above, the following representatives of company members of the Society passed away:

G. Worthen Agee, Barrow-Agee Laboratories, Inc.
James W. Ames, W. Ames and Co.
Russell Anderson, Belle City Malleable Iron Co.
John Henry Bateman, Louisiana State University
W. F. Carter, Acme Steel Co.
I. Chesler, Eagle Pencil Co., Inc.
A. E. Chester, Promat Division, Poor and Co.
Raymond Deutsch, Monarch Aluminum Manufacturing Co.
Grant J. Durant, Froehling & Robertson, Inc.
Guy C. Hecker, American Transit Assn.
James H. Holden, Pittsburgh Testing Laboratory
C. M. Houck, Pittsburgh Testing Laboratory
G. A. Lilliequist, American Steel Foundries
C. H. Masland, II, C. H. Masland and Sons
Cornelio Miechielsen, Técnica Industrial, S. A.
E. J. Morgan, Gilman Paper Co.
J. C. Morris, Oak Ridge National Laboratory
R. H. Patch, E. F. Houghton and Co.
Beauford H. Reeves, Rockbestos Products Corp.
C. C. Rose, Willard Storage Battery Co.
Ferdinand Schurman, Fish-Schurman Corp.

A number of these men had been affiliated with the Society for many years and rendered outstanding services, particularly through work on the technical committees. The various issues of the ASTM BULLETIN have included appropriate notices and appreciation of their work.

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* Honorary Member.

Honors and Awards

In order to allow sufficient time for the actual publication of all papers to be reviewed by the various award committees, it was agreed to allow two years to elapse between the presentation of papers and the conferring of awards therefor. Accordingly, because of the transition period, there will be no Dudley Medal, Richard L. Templin, Sam Tour, Sanford E. Thompson or C. H. Hogentogler Awards presented at this Annual Meeting. Starting with the 1957 Annual Meeting these awards will again be on an annual basis.

Recipients of other honors to be given at the 1956 Annual Meeting are indicated below:

Award of Merit:

L. D. Betz
H. A. Bright
Simon Collier
W. L. Fink
H. F. Gonnerman
B. W. Gonser
Fred Hubbard
R. P. Koehring
V. T. Malcolm
J. W McBurney
H. R. Wolf

Max Hecht Award (industrial water, sponsored by Committee D-19):

A. A. Berk, Chief, Industrial Water Laboratory, Eastern Experiment Station, Bureau of Mines, College Park, Md.

The *Harold DeWitt Smith Memorial Medal* (textiles) was presented to Arthur G. Scroggie, Manager, Characterization Laboratory, Textile Research Lab., E. I. du Pont de Nemours and Co., Inc., at the Spring Meeting of Committee D-13.

Presentation of Papers:

As a means of improving presentations of papers at the Annual Meeting, a trial system of rating the presentations has been in effect for the past several years. On the recommendation of the Administrative Committee on Papers and Publications, the Board has agreed to continue these ratings on a formal basis, and the persons receiving the three highest ratings for outstanding presentations will be given recognition by a letter from the President and in the ASTM BULLETIN.

Finances

Report for 1955:

Full details of the fiscal operations of the Society for 1955 are included in the report of the auditors appearing in Appendix I.

Operating Receipts:

Our expectations that 1955 would be a good year income-wise were more than realized. The estimated receipts of $732,000 actually developed to be $851,803. Of this amount, receipts from dues and entrance fees were $252,072 or 29.6 per cent; receipts from sales of publications were $521,496 or 61.2 per cent; and receipts from miscellaneous sources consisting principally of advertising, income from investments, and registration fees totaled $78,235 or 9.2 per cent.

It will be noted that receipts from membership fees are again under 30 per cent of the total for the third consecutive year.

Operating Disbursements:

Total disbursements were $851,350 which included $100,000 for the 1955 Book of Standards. This sum, together with $120,000 earmarked in previous years, is the reserve that enables us to cover the cost of this seven-part book. We build up a reserve over a three year period.

A total of $349,962 was spent for publications, which compares with $337,500 for the previous year. Expenditures for salaries, which included extra summer help and considerable overtime, reached $305,136. This figure represents 35.8 per cent of total disbursements. Cost of employee benefits, including a new major medical policy and covering new participants in the retirement plan, totaled $32,855 which represents 10.8 per cent of salary expenditures.

Balance Sheet:

On December 10 our total assets in General Fund were $736,154 compared to $683,646 a year ago. In all funds, General, Building, Medal, etc., total assets were $1,344,743 compared with $1,253,398 a year ago. It should be pointed out that in the Balance Sheet no account has been taken of the assets of the Society in the form of publications in stock. This is consistent with many years' practice in this respect and, of course, results in a more conservative statement. An inventory of the principal technical publications as of May 1, 1956, is summarized below:

Proceedings (All back copies from 1954)	2 370
1955 Book of ASTM Standards	
Part 1	6 402
Part 2	5 417
Part 3	4 803
Part 4	3 333
Part 5	3 532
Part 6	3 936
Part 7	3 798
Special Compilations of Standards	21 298
Selected Standards for Students	5 857
Symposiums and Special Reprints	133 120
Reprints of Standards (Approx.)	235 000
Radiographic Standards for Steel Castings	32
Radiographic Standards for Aluminum and Magnesium	33

Not included in inventory:

(1) Viscosity Temperature Charts; Hardenability Charts and Petroleum Charts
(2) Knock Test Manual and Supplement
(3) Marburg Lectures
(4) Cotton Yarn Boards

Building Fund:

The principal transaction affecting our Building Fund in 1955 was the sale of a portion of our lot on Cherry Street for about $15,000.

Assets in the Building Fund, which include the current Race St. Headquarters, the Cherry St. property, and the balance of the lot immediately adjoining this, total $306,927.

As liabilities, the Balance Sheet shows all contributions from members and from other ASTM funds, accumulated income, interest, proceeds from the lot sales in excess of the cost, and the loan from ASTM General Funds which enabled the procurement of the Cherry St. property. This amounted to $54,700 as of December 10, 1955. It is being amortized in the amount of $8700 yearly and should be wiped out accordingly in about 6 years.

Investments:

During the year, an important operating change was made in the handling of our investments through the creation of an over-all Investment Fund. To this were transferred on June 1, 1955, investments held by the various operating funds such as General, Building, Research, etc., with each of these operating funds owning a share of the Investment Fund in direct ratio to the securities and cash transferred. Each operating fund, therefore, will benefit to the extent that the over-all Investment Fund earns dividends and interest which are then prorated.

As of December 10, 1955, the book value or cost of the securities in the fund totaled $547,328; the market value was $732,640.

The total income for the Investment Fund holdings in 1955 was $27,432.

The following changes in investments during the calendar year 1955 are listed as a matter of record:

GENERAL FUNDS

Bought	*Cost*
Bonds	
$600 Amer. Tel. & Tel.	$600.00
Common Stocks	
100 sh. Acme Steel Corp.	$3 156.96
100 sh. American Bakeries	3 432.00
100 sh. American Can Co.	3 884.25
50 sh. American Cyanamid Co.	2 569.77
150 sh. American Stores Co.	7 608.33
75 sh. Canadian Pacific Railway	2 384.11
45 sh. Climax Molybdenum	3 106.50
25 sh. Crown Zellerbach	1 430.00
50 sh. Delaware Power & Light Co.	1 891.11
50 sh. Florida Power & Light	1 969.44
50 sh. General Electric Co.	2 509.65
75 sh. General Mills, Inc.	5 582.13
10 sh. General Motors (with script)	753.88
80 sh. Houston Lighting & Power	3 441.07
25 sh. International Nickel Co. of Canada	1 854.31
100 sh. Kennecott Copper	10 660.37
50 sh. Louisville & Nashville Railroad	4 154.59
100 sh. Niagara Mohawk Power	3 395.75
50 sh. Phila Electric Co.	1 969.19
50 sh. Square "D" Co.	2 362.37
50 sh. Standard Oil Co. of New Jersey	5 690.87
25 sh. Swift and Co.	1 291.17
25 sh. Vick Chemical Co.	1 398.37
175 sh. Electric Corp.	11 753.54
100 sh. Sears Roebuck & Co., Split 3 for 1	
50 sh. Republic Steel Corp., Split 2 for 1	
100 sh. Ohio Oil Corp., Split 2 for 1	
25 sh. B. F. Goodrich Co., Split 2 for 1	
75 sh. General Telephone Co., 50% stock dividend	
3 sh. Dow Chemical Co., Dividend	
Mutual Funds	
108 sh. Eaton Howard Balanced Fund	$2 048.76
73 sh. Fundamental Investors	946.08
170.3 sh. Commonwealth Investment Co.	1 444.63
283 sh. Broad Street Investment Co., 50% stock dividend	
2 573 sh. Eaton Howard Balanced Fund, Split 2 for 1	
1 011 sh. Fundamental Investing Co., Split 2 for 1	
Total Bonds and Common	88 849.73
Total Mutual Funds	4 439.47
Total	$93 289.20

Sold	*Gain or Loss* (−)
Bonds	
$5 000 Bond Walworth Co., 84½	−1 252.11
6 000 Bond Balto. & Ohio RR, Series G, 105	+275.00
500 Bond Balto. & Ohio RR, Series J, 100	−50.00
7 500 Bond "G" Bonds (matured)	
1 000 Bond U. S. Treasury 2⅞ % 1960–55 (matured)	
Stock	
75 Aluminum Co. of America, 112½	+5 633.58
1 Consumers Power Co., 47	+7.07
100 Gulf Oil Corp., 69¼	+4 077.43
50 Chicago Rock Island, pfd. (called), 105½	+842.48

During the early months of 1956, no major purchases were made for the Investment Fund, but the following list involving bonds called or receipt of stock dividends or using purchase options will bring up to date changes in the Investment Fund.

$1 500. Baltimore & Ohio "M" called March 1, 1956
30 sh. Allegheny Ludlum pfd $4.375 converted to
62 sh. Allegheny Ludlum—Common
62 sh. Allegheny Ludlum—split 2 for 1
115 sh. Carpenter Steel—split 2 for 1
1 sh. Gulf Oil—converted from 96/100th share and $3.41
102 sh. Sears Roebuck and Co.—Split 3 for 1, plus 1 per cent dividend and $17.44
135.533 sh. Commonwealth Investment—Dividend
122 sh. Eaton Howard Balanced Fund
81 sh. Fundamental Investors

1956 Budget

1956 is the year when we expect very heavy returns from the sale of the new Book of Standards. From the accompanying table entitled "Budget Summary" it will be seen that our total income is expected to reach $967,500, of which 62.3 per cent, or $604,000, will come from distribution of publications.

1956 BUDGET SUMMARY
Estimated Receipts

		Per cent
Dues and Entrance Fees	$256 000	26.5
Publications	604 000	62.3
Miscellaneous	107 500	11.2
TOTAL CURRENT RECEIPTS	$967 500	
Estimated Disbursements		
Publications	$359 000	38.4
Salaries	327 000	35.0
General Office Expenses	93 000	10.0
Technical Committee Expenses	13 500	1.5
Meetings	40 000	4.3
Headquarters Occupancy Expenses	40 000	4.3
Retirement, Health	38 000	4.0
Miscellaneous Expenses	23 000	2.5
TOTAL OPERATING DISBURSEMENTS	$933 500	

Unusual expenses are coming up, including provisions for the West Coast meeting, but the Finance Committee is hopeful that the total will not exceed $933,500, leaving a modest $34,000 favorable balance.

Investment Counsel:

In the operation of the Investment Fund, the Finance Committee, which includes the three past Presidents on the Board, together with three other Directors concerned with investments, has had the advice of a small Investment Advisory Council. However, with increasing problems and uncertainties it has been felt desirable also to retain professional counsel and the firm of Loomis, Sayles & Co., Inc., has been engaged for one year.

Administrative Matters

Revised Regulations Governing Board of Directors:

During the past year, several revisions were made in the regulations governing the Board of Directors to bring them up to date and to simplify some of the operations. As the Society's work has grown, there has been an accompanying additional load, for example, on the Finance Committee Chairman, and the Subcommittees of the Board have more problems to review.

Provisions that were modified include the appointment of subcommittees of the Board, the opening of checking and saving funds accounts, and the payment of bills. One change here eases the burden on the Finance Committee chairman of approving vouchers. Now none over $200 are submitted, but all bills continue to be listed on a summary schedule which he approves.

Copies of the revised regulations will be sent to any member on request. Beginning with 1955, they were not published in the Year Book to aid in curtailing the size of the publication.

Headquarters Staff:

During the year, as announced in the BULLETIN, two members of the Staff who had been serving as Assistant Technical Secretaries were designated Senior Assistant Technical Secretaries, namely, Messrs. L. C. Gilbert and J. W. Caum. Among Mr. Caum's several responsibilities are the maintaining of Staff contacts with numerous committees in the metals fields. Mr. Gilbert includes in his area many of the committees covering structural materials in the "C" and "D" committee groups.

Cement Reference Laboratory:

Since the technical assistant of Committee C-1 on Cement operates under a provision in the Regulations Governing Technical Committees which permits them to engage such a personnel with compensation clearing through Society headquarters, it is pertinent to note that Mr. John R. Dwyer, for many years technical assistant of Committee C-1, has been succeeded by Mr. J. R. Dise. Both men have been on the Staff of the National Bureau of Standards and among their responsibilities direct the Cement Reference Laboratory, a most interesting joint project of the Bureau and ASTM providing for the periodic examination of the equipment in laboratories undertaking testing work in this field. The January BULLETIN carried an interesting article concerning Mr. Dwyer's long-time services.

Cooperative Activities

Believing that one of the most important obligations the Society has is the coordination of standards and research work with other interested societies and associations and, particularly, preventing duplication of effort, the Officers and the Staff, together with the technical committee officers have maintained close relations with many other leading bodies. We have developed fuller cooperative relations with various additional groups, some of these activities being noted here.

American Leather Chemists Association:

A joint committee has been established with the American Leather Chemists Association to coordinate standards work, especially methods of testing in this field. Further news of this activity appears in the May BULLETIN.

Technical Association of the Pulp and Paper Industry:

To coordinate standardization work better in the field of paper and paper products, a joint committee of representatives of TAPPI and of ASTM Committee D-6 on Paper and Paper Products has been established. One of the objectives is to implement the exchange of information on projects being undertaken in the respective organizations. The committee also will assist in providing for cross-representation of personnel.

National Association of Corrosion Engineers:

This rapidly growing, relatively young association has many important activities under way which are of interest to various ASTM technical committees, especially in the metals field but also of concern to ASTM Committee D-19 on Industrial Water. While there is an excellent cross-over of memberships and committee personnel, it has been deemed desirable by the officers of both societies to insure a minimum of overlap, and accordingly a small liaison group of three representatives from each body is being designated to counsel on new and current activities. Research work being sponsored by NACE will unquestionably be of service to ASTM committees as they pursue standardization projects. Some of this research and surveys may be carried out jointly.

Corrosion Research Council:

Because of its great interest in corrosion work, both research and standardization activities, the Society has become a sponsor of the Corrosion Research Council. This was organized with the help of a grant from the Engineering Foundation with the objective of sponsoring investigative work of a nature that normally would not be undertaken individually by any of the societies concerned with corrosion work generally—for example, developing better information on some of the basic problems involved. ASTM, almost from its establishment, has taken an intensive interest in corrosion and several of the technical committees concentrate specifically in this area. Many standards and numerous valuable publications have been issued.

Joint Committee on Boiler Feed Water Studies:

Because of some problems in the operation of the Joint Committee on Boiler Feed Water Studies, for which ASTM along with several other societies was a sponsor, it was decided to concentrate sponsorship with the ASME, and, accordingly, ASTM relinquished its sponsorship but will plan to keep in close touch with this work. Committee D-19 on Industrial Water is much concerned with this whole area.

National Association of State Purchasing Officials:

Our Staff has conferred with officers of the National Association of State Purchasing Officials concerning the growing interest of that group in standards, and has offered our services.

Staff Activities:

In many of our joint and cooperative activities, the representatives of the respective headquarters staffs assume an important place. It is gratifying that our relations with the staffs of the organizations referred to above, and in fact with a great many of the other associations, are on a very friendly, constructive basis. The ASTM Staff men especially concerned with technical work are constantly in touch with the personnel of other bodies.

During the year, the Executive Secretary and other members of the Staff have spoken at various meetings, including several sponsored by local purchasing agents' associations. The Committee on Standardization of the National Association of Purchasing Agents is doing effective work in stimulating the use of recognized standards.

The above references are to some examples of important cooperative activities and contacts which constantly are being made. The Society stands ready at all times to cooperate closely with any other groups concerned with standards or with research in the materials field. With so much pressure on engineers and technical people, ASTM feels it is important, especially in these times, to use the efforts and talents of our committee members and officers in the most efficient manner. Accordingly, continuing stress is placed on avoiding duplication of activities.

Government Contacts:

The Society continues to maintain active concern with the work of the National Bureau of Standards. The officers and technical personnel of the Bureau continue to render outstanding service in many phases of our work, and many of the Society's committees are interested in the Bureau activities. Several Fellowships are sponsored by the Society at the Bureau and there are other important continuing joint activities such as the Cement Reference Laboratory.

While there has not been a recent meeting of the ASTM Advisory Committee to the Bureau, appropriate contacts have been made with Federal authorities concerning legislation which would implement the Bureau's important work in calibration and testing especially in the apparatus field and the development of important standards needed by industry.

We have maintained close relations

with the Department of Defense, especially those agencies concerned with standardization.

A most important development during the year was the establishment of an arrangement whereby ASTM agrees to furnish at special prices quantities of the Society's standards which are referred to in Federal documents or required in Government operations. This development came about because of the cooperative work between the Standards Division of the General Services Administration and ASTM in coordinating specifications and tests. It is believed much more can be done along these lines, and the Society is pledged to cooperate fully.

Engineering Education

Probably there is no society or organization which depends more for its continuing progress on engineers and technical men than does ASTM. Representatives of most of the engineering professions have served with distinction on our committees and the outstanding contributions of the Society and the reputation it has achieved have been due to the engineering talents which industry and government have made available. It is of vital importance that we have a continuing influx of mature technical men and women, and thus the Society is concerned with stimulating engineering training. Because the Society is not a professional group, it has not seemed wise to push any formal projects, but through personal conferences we have acquainted the other engineering societies with our great concern that adequate training in materials technology be a part of all engineering curriculums.

It is gratifying to note that in the report of the American Society for Engineering Education on engineering curriculums emphasis is given to the importance of adequate courses of study in the materials field. Considering the tremendous investments of American industry in materials, both from the producing and the consuming standpoint, it seems obvious that the well trained engineer should have a reasonably good working knowledge of the properties and tests of materials.

Furthermore, it is vital in our economy that adequate engineering talents be made available to carry on the voluntary efforts under way in so many societies and associations. One modest way in which ASTM interest is evidenced is through the growing number of student membership awards underwritten by members and ASTM Districts.

Respectfully submitted on behalf of the Board of Directors,

C. H. FELLOWS,
President.

R. J. PAINTER,
Executive Secretary.

June, 1956

APPENDIX I

REPORT OF THE AUDITORS FOR THE FISCAL YEAR JANUARY 1 TO DECEMBER 10, 1955

Philadelphia, December 23, 1955

Mr. R. J. Painter, *Executive Secretary*
AMERICAN SOCIETY FOR TESTING MATERIALS
Philadelphia, Pa.

Dear Sir:

We have examined the books and accounts of the American Society for Testing Materials for the period ended December 10, 1955, the date the Society closed its books for the year 1955. We did not make a detailed audit of all transactions, but made tests to the extent we considered appropriate in determining the accuracy of the accounts.

Cash on deposit in checking and savings accounts, and cash represented by certificates of deposit were verified by direct correspondence with the various depositories and reconciled with the cash records. Accounts receivable were not verified by correspondence with debtors. An investment fund was created by action of the Finance Committee at its meeting on September 20, 1955. The investments held by the funds, as well as certificates of deposits and savings accounts, were transferred as of June 1, 1955 to an investment fund, with each fund owning a share of this fund in direct ratio to the securities and cash transferred to the fund. We examined the securities held by this fund on December 27, 1955. Income for the period under review was verified by us and found to be properly recorded on the books of the Society. Investments in securities held by the investment fund at December 10, 1955 at an aggregate cost of $547,328.07 had a market value of $732,639.78.

We have prepared and submit herewith balance sheet as of December 10, 1955, statement of cash receipts and disbursements and operations for the period ended that date, and other supporting schedules which, in our opinion present fairly the financial position of the Society at December 10, 1955, and the results of its operations for the period ended that date.

Respectfully submitted,
John Heins & Co.

BALANCE SHEET AS OF DECEMBER 10, 1955

(Including Special and Designated Funds)

ASSETS

General Funds:				
Cash		$112 125.00		
Less Check No. 590 as of December 9, 1955 (drawn against estimated cost of 1955 *Proceedings* and 1955 Book of Standards)		81 500.00	$30 625.00	
Investment Fund Participation (at cost)			519 038.45	
Accounts Receivable			101 808.80	
Loans Receivable (Building Fund)			54 700.00	
Advance to Forest Products Laboratory			500.00	
Total Current Assets—General Funds			706 672.25	
Furniture and Fixtures (depreciated book value)			29 482.34	
Total Assets—General Fund				$736 154.59
Building Fund:				
Cash			8 942.28	
Investments Fund Participation (at cost)			20 245.28	
Land—Cost			68 459.42	
Building and construction costs		263 380.43		
Less accumulated depreciation		54 100.00	209 280.43	
Total Assets—Building Fund				306 927.41
Other Special and Designated Funds:				
Cash:				
ASTM Research Fund		8 202.31		
Dudley Medal and Marburg Lecture Fund	819.77			
Richard L. Templin Award Fund	76.74			
Sam Tour Award Fund	235.01			
F. E. Richart Award Fund	45.82			
Total Medal and Lecture Funds		1 177.34		
Committee Funds		91 753.12	101 132.77	
Investments Fund Participations (at cost):				
ASTM Research Fund		65 037.97		
Dudley Medal and Marburg Lecture Fund		6 706.25		
Richard L. Templin Award Fund		2 146.00		
Sam Tour Award Fund		1 670.24		
F. E. Richart Award Fund		1 012.26		
Committee Funds		96 772.45	173 345.17	
Total Assets, Special and Designated Funds				301 661.57
Total Assets				$1 344 743.57

LIABILITIES

General Funds:			
Advance Collections		$13 290.68	
Accounts Payable:			
Committee C-1		70.63	
Joint Committee on X-ray Diffraction		107 551.35	
Institute of Petroleum		3 246.88	
Total Current Liabilities—General Funds		124 159.54	
Life Membership Fund	$3 912.94		
Publication Fund	16 841.80		
Book of Standards Reserve	120 400.18		
Executive Retirement Reserve	13 073.60		
Retirement Fund Reserve	25 686.21		
Reserve for Depreciation of Investments	20 000.00		
Reserve for Additional Cost of Replacement of Headquarters Building	25 000.00		
Special Annual Meeting Entertainment Account	6 586.64		
	231 501.37		
Surplus	380 493.68	611 995.05	
Total Liabilities and Surplus—General Funds			$736 154.59
Building Fund:			
Contributions from Members		177 918.90	
Contributions from ASTM General Funds		65 389.57	
Notes Payable—General Funds		54 700.00	
Accumulated Income, Profit on Investments		8 918.94	
Total Building Fund			306 927.41
Other Special and Designated Funds:			
ASTM Research Fund:			
Principal	74 996.60		
Income	5 502.31	80 498.91	
Dudley Medal and Marburg Lecture Fund:			
Principal	6 706.25		
Income	819.77	7 526.02	
Richard L. Templin Award Fund:			
Principal	2 146.00		
Income	76.74	2 222.74	
Sam Tour Award Fund:			
Principal	1 670.24		
Income	235.01	1 905.25	
F. E. Richart Award Fund:			
Principal	1 012.26		
Income	45.82	1 058.08	
Committee Funds, Unexpended Balance		208 450.57	
Total Special and Designated Funds			301 661.57
Total Liabilities and Funds			$1 344 743.57

Comparison of General Funds for Fiscal Years 1951-1955 Inclusive.

At Close of Fiscal Year	Assets				Liabilities			
	Cash	Investments	Accounts Receivable	Furniture and Fixtures	Accounts Payable	Reserve for Book of Standards	Miscellaneous Funds	Surplus
1951	$164 497.75	$263 995.13	$77 239.16	$18 758.61	$72 990.86	$101 900.18	$90 536 48	$259 063.13
1952	200 537.56	282 493.36	63 050.85	22 475.95	86 506.97	101 900.18	114 326.31	265 824.26
1953	101 212.48	338 008.56	150 822.09	22 814.82	88 136.41	60 400.18	115 116.97	348 604.39
1954	130 344.62	391 988.43	131 653.09	29 660.19	124 687.30	120 400.18	111 554.22	327 004.63
1955	30 625.00	519 038.45	157 008.80	29 482.34	124 159.54	120 400.18	111 101.19	380 493.68

Committee Funds

Committee A-1 On Steel		$4 084.19
Committee A-5 On Corrosion of Iron and Steel		1 053.13
Committee B-3 On Corrosion of Non-Ferrous Metals and Alloys		2 718.32
Committee B-6 On Die-Cast Metals and Alloys		2 918.26
Committee B-8 On Electrodeposited Metallic Coatings		11.25
Committee C-1 On Cement		1 754.30
Committee C-1 Cement Reference Laboratory		(877.89)
Committee C-9 Sanford E. Thompson Medal Fund		996.80
Committee C-9 On Concrete and Concrete Aggregates		162.62
Committee C-15 On Manufactured Masonry Units		448.54
Committee C-16-On Thermal Insulating Materials		380.70
Committee C-18 On Natural Building Stones		363.56
Committee D-1 On Paint, Varnish, Lacquer and Related Products		656.22
Committee D-2 On Petroleum Products and Lubricants		1 473.15
Committee D-2 ASTM-IBM		956.07
Committee D-2 D.C.C Reference Fuel Account		28 816.49
Committee D-2 D.C.C National Exchange Group		(666.87)
Committee D-2 D.C.C Equipment Development Project		848.70
Committee D-4 On Road and Paving Materials		841.52
Committee D-5 On Coal and Coke		722.24
Committee D-7 On Wood		32.00
Committee D-7 Wood Pole Research Fund		21 338.28
Committee D-9 On Electrical Insulating Materials		2 506.56
Committee D-10 On Shipping Containers		61.89
Committee D-11 On Rubber and Rubber-like Materials		285.96
Committee D-12 On Soaps and Detergents		757.17
Committee D-13 On Textile Materials		1 457.24
Committee D-15 On Engine Antifreezes		43.00
Committee D-18—C. A. Hogentogler Award Fund		16.26
Committee D-19—Max Hecht Award Fund		16.59
Committee E-3 On Chemical Analysis of Metals		159.45
Committee E-4 On Metallography		2.50
Committee E-5-On Fire test-Small Scale Tunnel Test Research Fund		1 500.00
Committee E 9 On Fatigue of Metals		230.45
Committee E-14 On Mass Spectrometry		1 471.35
ASA Sectional Committee on Specifications for Cast Iron Pipe		1 419.65
Chicago District Council		3 478.04
Cleveland District		582.79
Detroit District		2 436.79
Ohio Valley District		4.50
Philadelphia District		465.77
Philadelphia District Special (Annual Meeting Account)		1 619.34
Southern California District		200.59
Pittsburgh District		501.80
Western New York—Ontario District		236.82
Advisory Committee on Corrosion		62 523.58
Administrative Committee on Ultimate Consumer Goods		234.44
Joint Committee on X-ray Diffraction, chemical analysis		52 248.87
Joint Committee on X-ray Diffraction, research associates		3 685.14
Joint Committee on X-ray Diffraction, IBM calculator		100.00
		207 278.12
Undistributed Profit on Sales Investments		1 173.45
Accounted for as follows:		
Cash in bank and on hand	$91 753.12	
Investments (at cost)	19 925.00	$208 450.57
Investment Fund Participation	96 772.45	$208 450.57

ASTM RESEARCH FUND

Principal Account

	Total	*Invested*	*Uninvested Cash*
Balance, December 11, 1954, as reported	$71 189.69	$63 014.26	$8 175.43
Receipts: Transfer from Income Account	34.39		34.39
Stock Dividends Transferred from Income	284.55	284.55	
Profit on Sales of Investments	787.97	787.97	
Transferred from General Funds	2 700.00		2 700.00
U. S. Savings Bonds, Series G (matured)		(1 500.00)[a]	1 500.00
Stock Purchased		753.88	(753.88)
Cash to Investment Fund		8 955.94	(8 955.94)
Balance, December 10, 1955	$74 996.60	72 296.60	$2 700.00
Investment Fund Participation	$65 037.97		
618 Sh General Motors Co. Common (Book Value)[b] (market value $29 818.50)	7 258.63	$72 296.60	

Income Account

Balance, December 11, 1954, as reported	$4 252.25	
Receipts: Interest and Dividends on Investments	3 274.03	
Profit on Sale of Investments	787.97	$8 314.25
Disbursements: Transfers to Principal Account	1 106.91	
Contribution to Committee E-14	400.00	
Printing, etc.	1 305.03	2 811.94
Balance, December 10, 1955		$5 502.31

F. E. RICHART AWARD FUND

Principal Account

Balance, December 11, 1954	$1 000.00		$1 000.00
Receipts: Transfer from Income Account	12.26	$12.26	
Transfer to Investment Fund		1 000.00	(1 000.00)
Balance, December 10, 1955	$1 012.26	$1 012.26	0

Income Account

Balance, December 11, 1954	$10.70	
Receipts: Interest and Dividends on Investments	35.12	
Profit on Sale of Investments	12.26	$58.08
Disbursements, Transfer to Principal Account		12.26
Balance, December 10, 1955		$45.82

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[a] ( ) Indicate red figures.

[b] Income from these shares is by agreement paid to an annuitant.

DUDLEY MEDAL AND MARBURG LECTURE FUND

Principal Account

	Total	*Invested*
Balance, December 11, 1954	$6 625.00	$6 625.00
Receipts, Transfer from Income (Gain on Sale of Investments)	81.25	81.25
Balance, December 10, 1955	$6 706.25	$6 706.25

Income Account

Balance, December 11, 1954, as reported	$896.56	
Receipts, Interest, Dividends and Profit on Investments	419.61	$1 316.17
Disbursements: Honorarium	400.00	
Miscellaneous	15.15	
Transfer to Principal Account	81.25	496.40
Balance, December 10, 1955		$819.77

RICHARD L. TEMPLIN AWARD FUND

Principal Account

			Uninvested Cash
Balance, December 11, 1954, as reported	$2 100.43	$1 088.49	$1 011.94
Receipts: Transfer from Income (Stock Dividend)	18.97	18.97	
Transfer from Income Account	26.60	26.00	
Transfer to Investment Fund		1 012.54	(1 011.94)
Balance, December 10, 1955	$2 146.00	$2 146.00	0

Income Account

Balance, December 11, 1954, as reported	$70.10	
Receipts: Interest and Dividends on Investments	96.91	
Gain on Sales of Investments	26.00	$193.01
Disbursements: Transfer to Principal Account	45.47	
Templin Awards	50.00	
Miscellaneous	20.70	116.27
Balance, December 10, 1955		$76.74

SAM TOUR AWARD FUND

Principal Account

Balance, December 11, 1954, as reported	$1 600.87	$1 600.87
Receipts, Transfer from Income Account	69.37	69.37
Balance, December 10, 1955	$1 670.24	$1 670.24

Income Account

Balance, December 11, 1954, as reported	$172.55	
Receipts: Interest and Dividends on Investments	111.59	
Profit on Sales of Investments	20.24	$304.38
Disbursements: Transfer to Principal Account	58.18	
Investment Account Participation	11.19	69.37
Balance, December 10, 1955		$235.01

RECEIPTS AND DISBURSEMENTS

FOR THE PERIOD DECEMBER 11, 1954 TO DECEMBER 10, 1955

Cash balance, December 11, 1954			$130 344.62
RECEIPTS			
Operating Receipts (Budgeted):			
Dues and Entrance Fees:			
Current Dues	$241 047.30		
Life membership	100.44		
Past Dues	1 063.50		
Advance Dues	4 224.57		
Income, Life Membership Fund	175.00		
Entrance Fees	5 461.00		
Total Dues		$252 071.81	
Sales of Publications:			
Book of Standards (Members, additional parts)	53 850.25		
1952 Book of Standards	58 800.25		
1955 Book of Standards	32 987.70		
1953 Supplements to Book of Standards	19 997.39		
1954 Supplements to Book of Standards	59 478.56		
Methods of Chemical Analysis of Metals	3 108.99		
Compilation of Standards	88 930.53		
Separate Standards	32 221.59		
Selected Standards for Students	3 966.10		
Proceedings	7 425.65		
50-Year Index and Papers	427.95		
BULLETIN Subscriptions (nonmembers)	3 981.68		
Special and Miscellaneous Publications	156 319.71		
Total Sales of Publications		521 496.35	
Miscellaneous:			
Advertising—BULLETIN	30 160.77		
Advertising—Index	1 833.50		
Interest and Dividends	27 162.18		
Registration and Other Fees—Meetings	13 701.11		
ASTM Exhibit at Annual Meeting	750.00		
Staff Services	4 059.30		
Miscellaneous	568.48		
Total Miscellaneous Items		78 235.34	
Total Operating Receipts (Budgeted)		851 803.50	
Nonoperating Receipts (Not Budgeted):			
Investments Matured or Sold	31 070.25		
Excess Remittance	3 625.62		
Committee C-1 for Technical Assistant	2 580.00		
Retirement Fund Reserve	1 502.05		
Joint AIME-ASTM Publication Fund	17.50		
Sale of X-ray Diffraction Cards	67 978.47		
British and IP Metric Tables	521.07		
Institute of Petroleum Publications	2 416.97		
Special Contribution Fund	600.00		
Furniture and Fixtures sold	50.00		
From Building Fund on Account of Loan	23 700.12		
Annual Meeting Luncheon and Buffet Supper	1 425.25		
Gain, Sale of Investments	16 849.01		
Gillett Memorial	401.25		
Investment Fund (Special Account)	6 426.85		
Miscellaneous	38.38		
Total Nonoperating Receipts		159 202.79	
Total Receipts			1 011 006.29
Total Receipts for 1955 and Cash Balance, December 11, 1954			**$1 141 350.91**

DISBURSEMENTS

Operating Disbursements (Budgeted):			
Publications:			
Year Book	$12 086.16		
1952 Book of Standards	2 196.38		
1955 Book of Standards	100 105.56		
1954 Supplements to Book of Standards	3 349.28		
Methods of Chemical Analysis of Metals	63.89		
Compilation of Standards	28 111.83		
Separate Standards	10 790.17		
Proceedings—repurchase of old numbers	186.50		
1954–1955 Proceedings	33 937.97		
Preprints	9 943.22		
ASTM BULLETIN	44 798.76		
Circulars to members	3 666.11		
Index to Standards	9 240.01		
Special and Miscellaneous Publications	91 487.10		
Total Disbursements—Publications		$349 962.94	
Salaries	305 362.30		
General Office Expenses	83 341.90		
Expenses—Technical and District Committees	10 430.15		
Expenses—Meetings	16 690.84		
Headquarters Occupancy Expense (includes $9 500 depreciation on building)	35 319.15		
Transfer to Princial of Research Fund	2 700.00		
American Standards Association	1 500.00		
Traveling Expense, Administrative and Special Committees	6 551.71		
Furniture and Fixtures	4 083.91		
Dues Contributions, Miscellaneous	1 303.96		
Legal Services	1 250.00		
Group Insurance	714.83		
Employees' Retirement Fund	23 868.76		
Federal Old Age Survivors	4 688.83		
Blue Cross	1 185.66		
Major Medical	2 396.88	501 388.88	
Total Operating Disbursements (Budgeted)		851 351.82	
Nonoperating Disbursements (Not Budgeted):			
Refund of Excess Remittances	3 613.12		
Investment Fund	167 831.28		
Technical Assistant Committee C-1	2 618.40		
Transfer to Joint Committee on X-ray Diffraction	65 000.00		
Cost and Refunds on X-ray Diffraction Cards	5 246.77		
Institute of Petroleum Publications	1 975.50		
Annual Meeting Luncheon and Buffet Supper	1 555.95		
Executive Retirement Reserve	900.00		
Employee Retirement Reserve	155.52		
Gillett Memorial Fund	450.15		
Commercial Directory	1 868.49		
Investment Fund (Special Account)	6 426.85		
Special Annual Meeting Account	1000.00		
Special Contribution	600.00		
Charge for Bad Debts	34.92		
Miscellaneous	97.14		
Total Nonoperating Disbursements		259 374.09	
Total Disbursements			1 110 725.91
Cash Balance, December 10, 1955			$30 625.00

ASTM BUILDING FUND

DETAILS OF ASTM BUILDING FUND CASH RECEIPTS AND DISBURSEMENTS FOR THE PERIOD DECEMBER 11, 1954 TO DECEMBER 10, 1955

Cash balance, December 11, 1954			$13 259.54
Receipts			
Contributions from Members		$2 155.41	
Interest and Dividends and Profit on Investments		622.94	
From ASTM General Fund:			
On Account of Depreciation on Building, Elevator, and Air Conditioning Installation		9 500.00	
On Account of Amortization of Note		8 700.00	35 752.34
From Sale of 1929 Cherry St. and 1930 Quarry St.		14 773.99	
			49 011.88
Disbursements			
Expenses of Cherry St. Property:			
Real Estate Taxes	$339.50		
Other Expense	175.00	514.50	
Renovation Cost of 1921–1925 Cherry St.:			
Stuccoing	2 663.00		
Contractor	1 728.61		
Automatic Fire Alarm System	1 110.24		
Repairs to Radiators	196.68		
Lumber	101.28		
Paint	22.63		
Venetian Blinds	22.22	5 844.66	
Construction Cost of 1916 Race St.:			
Contractor	2 250.38		
Automatic Fire Alarm System	966.60		
Air Conditioners	254.47		
Partitions	171.70	3 643.15	
Transferred to Investment Fund		6 367.17	
Reduction of Loan from ASTM General Fund		23 700.12	40 069.60
Cash balance, December 10, 1955			$8 942.28

ASTM BUILDING FUND

DETAILS OF ASSETS AND LIABILITIES OF BUILDING FUND AS OF DECEMBER 10, 1955

Assets

Cash in Girard Trust Corn Exchange Bank, Philadelphia, Pa.					$8 942.28
Investments Fund Participation					20 245.28
Property 1916–1918 Race St., Philadelphia, Pa:					
Cost:					
Land				30 000.00	
Building			$26 503.35		
Construction (Net of Salvage)		$132 081.72			
Automatic Fire Alarm System		966.60			
Proof Room		337.07			
Telephone Equipment Room		648.20			
Air Conditioner		254.47	134 288.06		
			160 791.41		
Accumulated Depreciation (Provided from General Funds)			48 525.00	112 266.41	
				142 266.41	
Property 1921–1929 Cherry St., and 1922–1930 Quarry St., Philadelphia, Pa:					
Cost:					
Land			38 500.00		
Less, Sale 1929 Cherry St. and 1930 Quarry St.		7 700.00	30 800.00		
Buildings (Net of Income)	$48 520.29				
Construction	62 898.28				
	111 418.57				
Less, Sale 1929 Cherry St. and 1930 Quarry St.		9 720.02			
		101 698.55			
Accumulated Depreciation (Provided from General Funds)		5 575.00	96 123.55	126 923.55	
Property 1919 Cherry St., Philadelphia, Pa:					
Cost			7 659.42		
Improvements			890.47	8 549.89	277 739.85
Total assets					306 927.41

Liabilities and Funds

Contributions from Members			$177 918.90	
Contributions from ASTM Funds			65 389.57	
Accumulated Income:				
Interest and Dividends on Investments		$6 865.35		
Profit on Disposal of Securities, net		321.55		
Profit on Sale of 1915–1918 Cherry St.		13 148.52		
Rental on Cherry St. Property		699.09		
		21 034.51		
Less Heating of Cherry St. Property	$812.50			
Interest on Loan from General Fund	4 010.96			
Real Estate Taxes on Cherry St.	2 885.16			
Water and Sewerage on Cherry St.	126.23			
Loss on Sale of 1929 Cherry St. and 1930 Quarry St.	2 646.03			
Moving Expense	1 309.69			
Appraisal	325.00	12 115.57	8 918.94	
Loan Payable to ASTM General Funds			54 700.00	
				$306 927.41

ASTM INVESTMENT FUND PARTICIPATIONS

AS OF DECEMBER 10, 1955

Cash	$165 300.83
Investments (at cost)	547 328.07
(Market value $732 639.78)	
Total assets	$712 628.90

Participations by funds, per cent:		Initial Participations	Net Profit Sale of Securities	Total Participation (At Cost)
General Fund	72.83433	$512 750.00	$6 288.45	$519 038.45
Committee Funds	13.57964	95 600.00	1 172.45	96 772.45
Research Fund	9.12649	64 250.00	787.97	65 037.97
Building Fund	2.84093	20 000.00	245.28	20 245.28
Medal & Lecture Fund	.94105	6 625.00	81.25	6 706.25
Templin Award Fund	.30114	2 120.00	26.00	2 146.00
Sam Tour Fund	.23437	1 650.00	20.24	1 670.24
Richart Fund	.14205	1 000.00	12.26	1 012.26
	100.00000	$703 995.00	$8 633.90	$712 628.90

ASTM INVESTMENT FUND

DETAILS OF INVESTMENT FUND CASH RECEIPTS AND DISBURSEMENTS FOR THE PERIOD JUNE 1, 1955 TO DECEMBER 10, 1955

Receipts

Cash transferred from:			
General fund	$100 134.80		
Committee funds	82 600.00		
Research fund	8 955.94		
Building fund	6 367.17		
Medal & Lecture fund	2 048.36	$200 106.27	
Bonds matured		6 800.00	
Interest and dividends		14 915.48	
Total receipts			$221 821.75

Disbursements

Purchase of securities	41 580.81	
Rebate to Richart fund	24.63	
Distributon to funds	14 915.48	
Total disbursements		56 520.92
Cash balance, December 10, 1955		165 300.83
Balance composed of:		
Demand account	6 739.34	
Savings accounts	108 561.49	
Certificates of deposit	50 000.00	$165 300.83

INVESTMENT FUND

AS OF DECEMBER 10, 1955

Investments

Par Value		Cost or Book Value	Market Value[a]
	Bonds		
$600	American Telephone & Telegraph Conv. Debs. 3⅞s 1967	$600.00	$786.75
5 000	American Tobacco Debs. 3s 1969	5 067.08	4 906.25
500	B. & O. R. R. Ref. & Gen. Mtg. 6s Ser. "J" 12/1/95 (called)		
1 500	B. & O. R. R. Ref. & Gen. Mtg. 5s Ser. "M" 3/1/96	1 511.25	1 546.65
6 000	B. & O. R. R. Ref. & Gen. Mtg. 5s Ser. "G" 1995 (called)		
4 000	Erie Railroad Company 4½s 2015	2 832.00	3 160.00
5 000	Pacific Gas & Electric Co. 1st & Ref. 3⅛s Ser "W" 12/1/84	5 150.78	4 937.50

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[a] All market values of securities are as of December 9, 1955.

Par Value	*Bonds*	*Cost or Book Value*	*Market Value*[a]
4 000	Socony Mobil Co. 2½s Debs. 6/1/76	3 899.85	3 620.00
5 000	Southern Natural Gas Co. 1st Mtg. Pipe Line S F 3⅛s 2/1/74	5 109.81	4 750.00
15 000	U. S. Treasury 2s 1954 (redeemed)		
2 000	U. S. Treasury 2½s 6/15/64–69	2 000.00	1 913.75
1 000	U. S. Treasury 2⅞s 1955/60 (redeemed)		
5 000	U. S. Treasury 2½s 3/15/65–70	5 000.00	4 781.25
8 500	U. S. Treasury 2½s 6/15/67–72	8 632.83	8 080.31
3 000	U. S. Treasury 2½s 12/15/67–72	3 000.00	2 851.88
15 000	U. S. Treasury 2½s 11/15/61	15 000.00	14 657.81
2 000	U. S. Treasury 3¼s 6/15/78–83	2 000.00	2 102.50
15 000	U. S. Treasury 2½s 8/15/63	14 943.75	14 568.75
7 500	U. S. Savings Bonds Ser. "O" 2½s (redeemed)		
30 000	U. S. Savings Bonds Ser. "G" 2½s 6/1/61	30 000.00	28 740.00
5 000	U. S. Savings Bonds Ser. "G" 2½s 1/1/62	5 000.00	4 775.00
5 000	U. S. Savings Bonds Ser. "G" 2½s 2/1/62	5 000.00	4 775.00
5 000	Walworth Co. 3¼s Conv. Debs. 5/1/76 (sold)		
	Total Bonds	114 747.35	110 953.40
Shares	*Preferred Stocks*		
30	Allegheny Ludlum Steel Corp. $4.375 Cum. Pfd.	3 037.34	4 050.00
100	American Airlines, Inc. 3½ per cent Cum. Conv. Pfd.	9 277.55	11 900.00
10	American Cyanamid 3¾ per cent Cum. Pfd. Ser. "C"	1 019.75	1 315.00
30	Armstrong Cork Co., 3.75 per cent Cum. Pfd. Ser. "A"	2 947.09	2 925.00
50	Chicago, Rock Island & Pacific R.R. Co. Ser. "A" (sold)		
30	Consolidated Edison Co. $5 Cum. Pfd.	3 286.08	3 270.00
1	Consumers Power Co. (sold)		
30	Endicott Johnson Corp. 4 per cent Pfd	2 937.50	2 940.00
50	Food Machinery & Chemical Corp. 3¼ per cent Cum. Conv. Pfd	4 750.10	6 200.00
30	General Motors Corp. $5 Pfd	3 755.80	3 720.00
60	Jones & Laughlin Steel Corp. $5 Cum. Pfd. Ser. "A"	5 386.73	6 285.00
30	Long Island Lighting Co. 4.35 per cent Ser. "E" Pfd	3 015.00	3 105.00
30	P. Lorillard Co. 7 per cent Pfd	4 380.00	4 207.50
30	Mississippi Power & Light Co. 4.36 per cent Cum. Pfd	3 075.00	3 030.00
30	Philadelphia Electric Co. 4.40 per cent Cum. Pfd	3 285.00	3 330.00
47	Standard Slag Co. 2nd Pfd	2 115.00	2 115.00
30	Westinghouse Electric Corp. 3.80 per cent Cum. Pfd. Ser. "B"	3 067.74	2 992.50
	Total Preferred Stocks	55 335.68	61 385.00
Shares	*Common Stocks*	*Cost or Book Value*	*Market Value*[a]
100	Acme Steel Co.	$3 156.96	$3 250.00
310	Allis Chalmers Mfg. Co.	13 820.16	20 653.75
100	Aluminum Company of America (sold)		
100	Aluminum Company of America	2 061.45	8 025.00
100	American Bakeries Co.	3 432.00	3 450.00
100	American Can Co.	3 884.25	4 762.50
150	American Cyanamid Co.	6 238.91	9 918.75
100	American Radiator & Standard Sanitary Co.	1 564.32	2 225.00
150	American Stores Co.	7 608.33	8 550.00
50	American Telephone & Telegraph Co.	8 031.72	8 962.50
100	American Tobacco Co.	7 370.97	8 125.00
30	Atchison, Topeka & Sante Fe R.R. Co.	2 961.06	4 680.00
40	Bethlehem Steel Corp.	2 095.99	6 770.00
75	Canadian Pacific Railway Co.	2 384.11	2 606.25
115	Carpenter Steel Co.	6 104.75	9 573.75
125	Chase National Bank	3 412.50	6 421.88
100	Climax Molydenum Co.	5 409.11	6 662.50
100	Crown Zellerbach Corp.	2 914.79	5 600.00

Shares	*Common Stocks*	*Cost or Book Value*	*Market Value*[a]
67	Delaware Light & Power Co.	2 174.11	2 663.25
163	Dow Chemical Co.	3 944.32	9 372.50
30	E. I. du Pont de Nemours & Co	2 899.12	6 870.00
50	Florida Power & Light Co.	1 969.44	1 868.75
125	General Electric Co.	4 311.15	6 828.13
75	General Mills, Inc.	5 582.13	5 287.50
225	General Telephone Corp.	3 312.82	9 309.38
50	B. F. Goodrich Co.	1 647.31	3 962.50
128	Gulf Oil Corp.	3 473.27	10 928.00
100	Gulf Oil Corp. (sold)		
206	Houston Lighting & Power Co.	7 112.18	9 270.00
200	International Harvester Co.	6 406.67	7 625.00
125	International Nickel Co. of Canada, Ltd.	5 788.01	10 250.00
50	Johns-Manville Corp.	3 433.25	4 381.25
100	Jones & Laughlin Steel Corp.	2 636.06	5 150.00
100	Kennecott Copper Corp.	10 660.37	11 725.00
200	Lone Star Cement Corp.	4 941.44	14 400.00
50	Louisville & Nashville R.R. Co.	4 154.59	4 475.00
100	Montgomery Ward & Co.	5 798.04	9 537.50
103	Niagara Nohawk Power Corp.	3 479.57	3 424.75
200	Ohio Oil Co.	2 623.35	6 975.00
57	Pacific Gas & Electric Co.	2 165.63	2 850.00
50	Parke, Davis & Co.	1 641.19	2 056.25
50	Pennsylvania Power & Light Co.	2 083.20	2 437.50
50	Chas. Pfizer & Co., Inc.	1 732.94	2 043.75
321	Philadelphia Electric Co.	8 599.20	12 799.88
125	Public Service Electric & Gas Co.	4 558.50	4 250.00
100	Republic Steel Corp.	2 152.93	5 050.00
50	Sears, Roebuck & Co.	3 137.78	5 475.00
100	Socony Mobil Oil Co.	2 510.28	6 150.00
50	Southern Pacific Co.	2 234.45	2 887.50
50	Square D Co.	2 362.37	2 762.50
90	Standard Oil Co. of N. J.	8 526.48	13 387.50
25	Swift & Co.	1 291.17	1 212.50
40	The Texas Co.	2 184.35	4 760.00
60	Union Carbide & Carbon Corp.	4 235.24	6 660.00
25	Union Pacific R.R. Co.	2 719.78	4 662.50
30	United Gas Improvement Co.	446.68	1 053.75
25	Vicks Chemical Co.	1 398.37	1 368.75
240	Westinghouse Electric Corp.	15 644.26	14 100.00
	Total Common Stocks	238 423.38	360 507.77
Shares	*Mutual Funds*		
849	Broad Street Investing Corp.	11 337.87	18 482.73
3 871.398	Commonwealth Investment Co.	27 444.91	35 462.01
5.254	Eaton Howard Balanced Fund	80 823.46	111 805.12
2.095	Fundamental Investors, Inc.	19 215.42	34 043.75
	Total Mutual Funds	138 821.66	199 793.61
	Summary		
	Bonds	114 747.35	110 953.40
	Preferred Stocks	55 335.68	61 385.00
	Common Stocks	238 423.38	360 507.77
	Mutual Funds	138 821.66	199 793.61
		$547 328.07	$732 639.78

APPENDIX II

REPORT OF ADMINISTRATIVE COMMITTEE ON STANDARDS

The Administrative Committee on Standards is interested in all aspects of the Society's standardization work. The several functions are discussed under the subdivision headings of this report.

The review of recommendations from technical committees is, of course, one of the important functions of the Standards Committee, since in its establishment it was intended to provide a means of having recommendations acted upon in the interval between Annual Meetings. Most of the annual reports refer to recommendations that have been submitted to the Standards Committee during the year. The actions taken by the Standards Committee are summarized below:

New Tentatives	19
Revised Tentatives	38
Tentative Revisions of Standards	9
Standards Revised and Reverted to Tentative	5
Tentatives Withdrawn	1
Standards Withdrawn	3
Emergency Alternates Revised	1
Emergency Alternates Withdrawn	1

A large number of these recommendations were acted upon at a meeting held on September 15, 1955; the remainder were handled by correspondence.

Actions at 1955 Annual Meeting:

As a matter of record, the actions taken by the Society at the 1955 Annual Meeting with respect to standards and tentatives are tabulated below:

Existing Tentatives Adopted as Standard	92
Standards Revised	141
New Tentatives	65
Standards Revised and Reverted to Tentative	27
Tentative Revisions of Standards	19
Tentatives Revised	161
Standards and Tentatives Withdrawn	15

Form of Standards:

Improvement of the quality and form of standards continues to be an important consideration of the committee. Editorial committees have been organized by a number of the technical committees, and the Standards Committee would wish to see this practice extended. The recommendations covering the form of ASTM specifications referred to in previous reports as being in preparation have now been drafted and have been brought to the attention of all the technical committees. This document has had a very favorable reception and it is felt it should simplify considerably the work of the committees. A comparable manual covering recommendations on the form of methods of chemical analysis has recently been developed, and it is expected Committee E-1 on Methods of Testing will shortly have available a companion manual on methods of testing in general.

EXPANSION OF STANDARDIZATION ACTIVITIES

Many of the proposals dealing with new projects referred to in the report of the Standards Committee a year ago have now been acted upon favorably by the Board or have reached the point where recommendations are now under consideration for organizing work in these fields.

Electronic Materials.—A new Committee F-1 has been established to handle work in this field. Some of this work was formerly under the jurisdiction of a subcommittee on metallic materials for radio tubes and incandescent lamps of Committee B-4, but its importance was recognized as being such as to warrant a separate committee. The work is being expanded to include luminous materials and other phases that are closely related to the work.

Hydraulic Fluids.—The considerations referred to a year ago resulted in the reference of this subject to Committee D-2 on Petroleum Products and Lubricants, and a new technical committee has been established under Committee D-2 which will handle not only hydraulic fluids having a petroleum base but the synthetic fluids as well.

Sorbent Mineral Materials.—Based upon the recommendations of a conference called for the purpose of considering the undertaking of work in this field, the Board of Directors now has before it the recommendation that a new Committee on Sorbent Mineral Materials be established. This will include not only materials of the clay type but diatomaceous materials as well.

Carbon Black.—The need for methods of testing carbon black has been recognized by a group functioning in this field which has resulted in the development of a number of methods of testing. These have been brought to the attention of the Society with the suggestion that a new committee on carbon blacks be established, and such a committee has been authorized by the Board of Directors and is now in process of organization. The work will need to be coordinated with that of the several existing technical committees, as for example, Committee D-1 on Paint which is interested in carbon black as a pigment; Committee D-20 on Plastics which has work under way on carbon black as a filler or pigment; Committee D-11 on Rubber which has a group at work on the evaluation of carbon black as a reinforcement in rubber formulations.

Manganese Dioxide.—A group of those in the industry has been working on standards for manganese dioxide for use in dry batteries. A new ASTM committee on manganese dioxide is suggested.

Leather.—The discussions referred to a year ago with respect to setting up a committee on the physical testing of leather have culminated in the establishment of a joint committee with the American Leather Chemists Assn. In view of the extensive amount of work that has already taken place in the latter organization, it is expected excellent progress will be made in the establishment of methods that will receive the joint approval of the two organizations.

Casein.—On the recommendation of a conference called specifically for the purpose of reviewing the proposal, a new technical committee on casein has been authorized by the Board of Directors, having the several applications in mind, namely, paints, adhesives, and its use in paper. Representation is being secured from the several committees concerned. While the work will initially be confined to casein, it is possible that it will later be expanded to include other proteins.

Industrial Chemicals.—The subject of industrial chemicals is so broad that a preliminary conference should first be held to delineate the field more specifically. A number of projects undertaken during recent years, either in new committees or by existing technical committees, cover certain portions of this general field, so it would be well to determine more definitely what is still needed.

Magnet Wire.—The interest in magnet wire has increased considerably during the past few years, particularly in view of the introduction of a number of newer types of insulation. The Society has been represented on the Sectional Committee on Magnet Wire (C9), functioning under the sponsorship of the National Electrical Manufacturers Association. In addition to this, a new subcommittee has been established in ASTM Committee D-9 on Electrical Insulating Materials to deal with the methods of testing magnet wire, and the representation of the Society on the sectional committee has been increased in order to tie in this new work effectively with the work of the sectional committee. It is not proposed to carry out work on specifications for magnet wire independent of the sectional committee. Specifications for the conductor for use in magnet wire are covered by ASTM Committee B-1.

Cooperative Relations.—To an ever increasing extent, there is an interlocking of interest among the technical committees, particularly in the nonmetallic field. More recently the question has developed as to how the Society might best handle the subject of foamed plastics. Committee D-11 has been quite active in dealing with the rubber-like foams, whereas a number of committees are interested in various applications of the rigid plastic foams. A coordinating committee has been set up to include representation from the various committees concerned.

RELATIONS WITH AMERICAN STANDARDS ASSOCIATION

Two methods under the procedure of the American Standards Association are used by ASTM in submitting recommendations to the ASA. One is a combination of the so-called existing standards procedure and the proprietary sponsorship procedure. On the initial reference of an existing ASTM standard to the ASA, it is submitted under the existing standards procedure with a supporting statement giving a history of the development of the standard and indicating the degree of its acceptance. If the standard is approved as American Standard, the ASTM is granted proprietary sponsorship insofar as any future revisions are concerned, and such revisions are brought to the attention of the ASA currently for approval under this proprietary sponsorship procedure.

The second method consists of having standards reviewed in a sectional committee made up of representatives of a number of interested organizations, usually with the ASTM as sponsor or cosponsor of the sectional committee.

Standards Submitted Under the Existing Standards Procedure:

The following standards were submitted to the American Standards Association for approval as American Standard under the existing standards procedure: 36 standards dealing with refractories, 14 standards dealing with electrodeposited metallic coatings, and 2 standards dealing with fire tests of building constructions.

Revised Standards Submitted Under the Proprietary Procedure:

In view of the Society having been designated as proprietary sponsor for the revisions of ASTM standards that had been approved as American Standard under the existing standards procedure, it has submitted to the American Standards Association during the year revisions of 20 standards relating to steel, 1 relating to gray-iron castings, 2 relating to corrosion of iron and steel, 5 relating to copper and copper alloys, 1 relating to drain tile, 2 relating to gypsum, 6 relating to manufactured masonry units, 1 to aggregate for interior plastic, 3 to pigments for paint, 2 to linseed oil, 1 relating to gaseous fuels, 1 relating to rubber, 5 relating to soaps and detergents, 1 relating to thermometers, and 2 relating to fire tests of building constructions.

Standards Submitted Under the Sectional Committee Procedure:

During the year, the following five sectional committees, for which the Society is sponsor or cosponsor, took action to present recommendations to the ASA as follows:

Sectional Committee C7 on Bare Electrical Conductors.—This committee has recommended the approval as American Standard of 3 ASTM standards, and the approval of revisions of 13 American Standards.

Sectional Committee C59 on Electrical Insulating Materials.—This committee has recommended the approval as American Standard of 1 ASTM standard, and the approval of revisions of 5 American Standards.

Sectional Committee B36 on Wrought-Iron and Wrought-Steel Pipe and Tubing.—This committee has recommended the approval as American Standard of 16 ASTM standards, and the approval of revisions of 16 American Standards.

Sectional Committee L14 on Textile Test Methods.—This committee has recommended the approval as American Standard of 20 standards prepared by the American Association of Textile Chemists and Colorists, certain of which are identical with ASTM standards, the approval of revisions of 18 American Standards, and the withdrawal of 2 American Standards.

Sectional Committee Z11 on Petroleum Products and Lubricants.—This committee has recommended the approval as American Standard of 5 ASTM standards, and the approval of revisions of 13 American Standards.

INTERNATIONAL RELATIONS

The Society continues to receive from other countries standards with the request for comment. ASTM committees review these standards and frequently suggestions result. Apart from such informal cooperation, however, quite a number of proposals are received through the American Standards Association concerning projects that have been organized under the International Organization for Standardization (ISO). The various ISO projects of interest to ASTM are listed below:

Textiles.—Great Britain holds the general secretariat for ISO/TC 38 on Textiles, but the United States holds the secretariat for several subcommittees. Participation by the United States in this work is being handled by a special committee organized under the sponsorship of ASTM and the American Association of Textile Chemists and Colorists, with representation from the various groups interested. This special committee has been given the designation "L23."

Petroleum Products.—The United States holds the secretariat for ISO/TC 28 on Petroleum Products, the technical work of the secretariat being under the purview of Sectional Committee Z11, for which ASTM is sponsor.

Rubber.—The secretariat for ISO/TC 45 on Rubber is held by Great Britain. The group to handle United States participation in this ISO committee has been set up under ASTM Committee D-11.

Viscosity.—The United States holds the secretariat for ISO/TC 66 on Viscosity. The Subcommittee on Rheological Properties of ASTM Committee E-1 has been set up to handle the work of the secretariat.

Plastics. –The United States holds the secretariat for ISO/TC 61 on Plastics. ASTM Committee D-20 on Plastics is handling the work of the secretariat and has organized for the purpose a special committee consisting of representatives of the various groups interested.

Shellac.—Subcommittee XIII on Shellac of ASTM Committee D-1 has been set up as the committee in charge of contacts by the United States with ISO/TC 50 on Lac, for which India holds the secretariat.

Solid Mineral Fuels.—ASTM Committee D-5 is handling the contacts of the United States with ISO/TC 27 on Solid Mineral Fuels, for which Great Britain holds the secretariat.

Iron and Steel.—The United States has the status of an observer in the work of ISO/TC 17 on Iron and Steel for which Great Britain holds the secretariat. A number of draft proposals have been received which have been brought to the attention of Committee A-1 on Steel, while others dealing with methods of testing have been submitted to the appropriate subcommittees of Committee E-1.

Chemistry.—Italy holds the secretariat for ISO/TC 47 on Chemistry. Several ASTM committees have indicated an interest in this work, and draft proposals which have been received have been passed on to the committees.

Laboratory Glassware.—Great Britain holds the secretariat for ISO/TC 48 on Laboratory Glassware. The United States is participating in this work through a special group on which are represented ASTM Committee E-1 and the Scientific Apparatus Manufacturers Association.

Statistical Treatment of Series of Observations.—ASTM Committee E-11 on Quality Control favors participation by the United States in the work of ISO/TC 69. This recommendation is concurred in by the American Society for Quality Control, American Statistical Association, and Institute of Mathematical Statistics.

Mica.—Subcommittee IX on Mica of ASTM Committee D-9 has been set up to handle the United States contacts with ISO/TC 56 on Mica, for which India holds the secretariat.

Copper and Copper Alloys.—The United States holds the secretariat for ISO/TC 26 on Copper and Copper Alloys. The advisory Committee of ASTM Committee B-5 has been set up to handle the work of the secretariat.

Paper.—On the recommendation of ASTM Committee D-6, the United States participates in the work of ISO/TC 6 on the basis of an observer. France holds the secretariat for this ISO committee.

Timber.—ASTM Committee D-7 on Wood has indicated that it favors United States participation in the work of ISO/TC 55 on Timber, for which Russia holds the secretariat.

Raw Materials for Paints, Varnishes, and Similar Products.—ASTM Committee D-1 has indicated that it favors United States participation in the work of ISO/TC 35 on Raw Materials for Paints, Varnishes, and Similar Products.

Asbestos-Cement Products.—ASTM Committee C-17 on Asbestos-Cement Products has indicated that it favors United States participation in the work of ISO/TC 77 on the basis of an observer. Switzerland holds the secretariat for this ISO committee.

Light Metals and Their Alloys.—ASTM Committee B-7 has indicated that it favors United States participation in the work of ISO/TC 79 on Light Metals and Alloys.

Refractories.—ASTM Committee C-8 has indicated that it favors United States participation in the work of ISO/TC 33 on Refractories.

Aromatic Hydrocarbons.—ASTM Committee D-16 has indicated that it favors United States participation in the work of ISO/TC 78 on Aromatic Hydrocarbons for which Great Britain holds the secretariat.

Cast Iron.—Great Britain holds the secretariat for ISO/TC 25 on Cast Iron. The Advisory Committee of ASTM Committee A-3 has been set up to handle United States participation.

Respectfully submitted on behalf of the committee,

J H. FOOTE,
Chairman.

R. E. HESS,
Secretary.

APPENDIX III

REPORT OF ADMINISTRATIVE COMMITTEE ON RESEARCH

One meeting of the Administrative Committee on Research was held during the past year—at ASTM Headquarters on October 3, 1955.

Two new members of the committee were appointed: W. H. Lutz and H. K. Nason. It is with regret that the loss of two valued members is reported, H. H. Lester by death and O. W. Ellis by resignation because of retirement.

A special subcommittee, of which Dr. Lester was chairman, has reviewed in detail the status of the ACR activity in publicizing "unsolved problems" of interest to ASTM technical committees. It was the feeling that this activity really did serve a useful purpose and should be continued. The close cooperation of ASTM technical committees in suggesting and following the progress in solving needed researches is being sought. This subcommittee is being continued, under the chairmanship of A. T. McPherson, to implement the recommendations that have been made to improve this project.

Short articles were written by members of the Administrative Committee throughout the year for most issues of the BULLETIN under "ACR Notes." Also, an article on "Materials Research in ASTM" was written on behalf of the committee by Dr. Lester. A "Review of ASTM Research" for the period of December, 1952, to December, 1955, has been planned, which is a continuation of previous reviews.

The ACR is cooperating with Committee B-2 on Non-Ferrous Metals and Alloys in sponsoring a symposium on titanium to be given at the West Coast Meeting in September, 1956. This is aimed at presenting papers and discussion on properties of titanium and its alloys which affect mechanical performance and on factors affecting methods of testing. Tentative plans are being made to sponsor a symposium on materials research at the 1958 Annual Meeting of ASTM in Boston.

Active interest and encouragement in technical committee research projects has continued. In particular, researches involving the solicitation of funds have been followed closely. These include the ASTM-ASME Joint Committee on Effect of Temperature on Properties of Metals, the Joint Committee on Chemical Analysis by Powder Diffraction Methods, Committee C-16's research on Thermal Insulating Materials (under contract with the Pennsylvania State University), Committee D-7's research on wood the testing of wood poles (under contract with the U. S. Forest Products Laboratory), Committee E-5's research on Fire Tests (under contract with the U.S. Forest Products Laboratory), and the Advisory Committee on Corrosion. Research funds involved in these activities total about $375,000.

Representation by members of the ACR is maintained on the National Research Council, Division of Industrial and Engineering Research, and with the Engineering Foundation research activities. At its last meeting, the ACR recommended that ASTM support the Engineering Foundation Corrosion Research Council, independently of the Inter-Society Corrosion Committee.

Financially, the Committee's Research Fund has been increased moderately during the past year. This was particularly aided by $7271.90 bequeathed by H. J. Love. The balance of the Principal Account as of the end of the year was $74,996.60. The balance of the Income Account was $5502.31. The Income Account does not permit the substantial financing of research projects. However, it does permit financing some minor projects, aid to some needy, separately financed researches, and the encouragement or stimulation of researches.

Respectfully submitted on behalf of the committee,

BRUCE W. GONSER,
Chairman.

J. S. PETTIBONE,
Secretary.

APPENDIX IV

REPORT OF ADMINISTRATIVE COMMITTEE ON PAPERS AND PUBLICATIONS

The year 1955 was naturally a very heavy publication year by virtue of the appearance of the Book of Standards which alone overshadows all other publication ventures. But apart from this, the committee was confronted not only with the planning for the 1956 Annual Meeting but the supervision of the program for the Pacific Coast Area meeting to be held in September in Los Angeles. In addition to this, a Symposium on Properties, Tests, and Performance of Electrodeposited Metallic Coatings was held in connection with the Buffalo Committee Week, and various papers sessions have been sponsored by individual committees in connection with the meetings of these committees throughout the year. Many of the papers presented are subsequently brought to the attention of the Papers Committee for publication in the ASTM BULLETIN or in the *Proceedings*.

West Coast Meeting:

In organizing for the West Coast meeting, a technical program committee was set up under the General Committee on Arrangements. This program committee sought the assistance of various groups on the Pacific Coast in the development of the program for the Los Angeles meeting. In addition to this, the technical committees of the Society were approached both with respect to holding meetings in conjunction with the West Coast meeting and also with respect to sponsoring technical programs. Quite a few of the sessions of the West Coast meeting accordingly are committee sponsored. Committee D-2 on Petroleum Products and Lubricants for example has organized three symposiums—Vapor Phase Oxidation of Gasoline, Turbine Oils, and Lubricating Oils. By way of operation, symposium or session committees have been set up responsible for the individual programs, with representation from both the West Coast Technical Program Committee and from the Administrative Committee on Papers and Publications. These groups serve not only in organizing the sessions but also have the responsibility for the review of the papers as to their acceptability both for presentation and subsequent publication. In number of sessions (44) and in number of papers, the West Coast meeting will probably be the largest ever sponsored by the Society.

1956 Annual Meeting:

The program in prospect for the 1956 Annual Meeting includes the following symposiums:

Symposium on Solder
Symposium on Specific Gravity of Bituminous Coated Aggregates
Symposium on In-Place Shear Testing of Foundation Soil by the Vane Method
Symposium on pH Measurement
Informal Symposium on Rheology

Symposium on Tension Testing of Non-Metallic Materials
Symposium on Ion Exchange and Chromatography in Analytical Chemistry
Symposium on Steam Quality

1955 Book of ASTM Standards:

The 1955 Book of ASTM Standards totals close to 12,000 pages, in seven parts, the edition for the individual parts ranging from 10,400 to 19,500 copies. The press work was such that two presses were kept running continuously and for a greater part of the time three presses from early in September until the end of January. While in the report a year ago it was indicated that some of the press work might be handled by a second printer, our regular printer was able to handle it through the installation of an additional press. It was, however, necessary to have two of the parts folded and bound at a second bindery, and it was only in this way that the books were made available as expeditiously as was the case. A special thin paper was used having greater opacity than that of the previous edition but without increasing the bulk materially.

1955 Proceedings:

The Papers Committee has been asked by the Board to review the *Proceedings* and their distribution with the thought that perhaps the annual cost of the *Proceedings* could be reduced in some way. There is the possibility of splitting the *Proceedings*, and this has been proposed to provide members with only those portions in which they would have an interest. This could be either according to material lines or, according to one suggestion, by dividing the *Proceedings* according to reports of committees and papers. This latter would introduce a complication that many of the committee reports have appended thereto technical papers or the equivalent type of material published as subcommittee reports or as special reports, and accordingly it would not be easy to segregate purely report material if by this is meant the report of the activities of the committee and the record of its recommendations dealing with standards. Dividing the *Proceedings* according to materials would also be difficult and if carried into too detailed a breakdown would result in such limited editions of each as to be uneconomical.

In order to secure some idea of the interest in the *Proceedings*, they are being published in the same manner as heretofore but with selective distribution so that each member will need to request a copy if interested in receiving it.

1955 Year Book:

Also as an economy measure, since the Year Book has been increasing so greatly in size due to the increase in the membership list and more particularly in the committee personnel, a number of items heretofore appearing in the Year Book have been eliminated, as for example the geographical distribution, the regulations governing technical committee, the regulations governing the Board of Directors, etc. The regulations are available separately. A considerable saving in size was effected in this way. The Year Book, however, will need to be given further study in the committee. Perhaps the more essential information concerning the committee personnel could be given more effectively without any increase in size.

Other Publications:

In addition to the Book of Standards, a number of compilations of standards were issued as indicated below. In so far as possible, these compilations were made as overruns, either in part or in substantial entirety, of the Book of Standards, which accounts for the special pagination in these compilations. This naturally effects considerable economy in the productions of these books. The appearance of the annual *Proceedings* was somewhat delayed in deference to concentration on the publication of the Book of Standards, but the *Proceedings* have been completed and are now in press as well as a large number of special technical publications covering the various symposiums held at the 1955 Annual Meeting. It is expected that all of these will be available before the current annual meeting. The special volume covering the Significance of Tests of Concrete and Concrete Aggregates referred to in last year's report, comprises some 390 pages and became available in the fall of the year. Other special publications were issued as listed below.

Presentation of Papers:

For the past few years, an effort has been made to improve the character of the presentation of papers. A means of rating presentations has been set up which, on a trial basis, has proved successful. The rating scheme is one in which reporters at each of the sessions, by means of check marks and one-word replies, complete a comprehensive statement on items usually concerned with effective presentation. From these the nature of the presentation can be judged by a reviewing committee. The scheme has already been effective in enlisting the interest of authors in making better presentations, and considerable interest has also been aroused in other quarters on the efforts that the Papers Committee is making. The feasibility and value of the plan was amply demonstrated, so this year recognition is being made of the best presentations at the 1955 Annual Meeting.

Record of Publications Issued This Year

Regular Publications:

1955 Proceedings, 1250 pp., 6500 copies.
1955 Book of Standards
  Part 1, 1836 pp., 19,500 copies.
  Part 2, 1516 pp., 15,400 copies.
  Part 3, 2062 pp., 13,000 copies.
  Part 4, 1426 pp., 10,400 copies.
  Part 5, 1494 pp., 10,600 copies.
  Part 6, 1776 pp., 10,800 copies.
  Part 7, 1642 pp., 10,400 copies.
1955 Year Book, 620 pp., 5750 copies.
1955 Index to Standards, 216 pp., 23,000 copies.
ASTM Bulletin, 8 issues (May 1955 to April 1956) total number of pages 808, average number of copies 13,400 (244 pages of technical papers).

A statement of the volume of the principal publications for the past four years is given below:

	1952 to 1953 Pages	1953 to 1954 Pages	1954 to 1955 Pages	1955 to 1956 Pages
Proceedings	1335	1209	1425	1250
Book of ASTM Standards	9976	....	....	11 752
Supplements to Book of Standards	....	2099	2746	....
ASTM Methods of Chemical Analysis of Metals	....	....	....	....
Index to ASTM Standards	300	300	306	216
Year Book	632	644	672	620
Special Technical Publications	1606	2604	2101	3018
Other Special Publications	1182	938	46	964
ASTM Bulletin	716	724	792	808
Totals	15 747	8518	8088	18 506

Special Compilations of Standards Published from April 15, 1955 to April 15, 1956:

Specifications for Steel Piping Materials (A-1), 435 pp., 3000 copies.

Standards on Metallic Electric Conductors (B-1), 308 pp., 800 copies.

Standards on Copper and Copper Alloys (B-5), 656 pp., 1500 copies.
Standards on Light Metals and Alloys (B-7), 291 pp., 800 copies.
Standards on Electrodeposited Metallic Coatings (B-8), 103 pp., 5000 copies.
Standards on Cement (C-1), 278 pp., 2500 copies.
Standards on Mineral Aggregates, Concrete and Nonbituminous Highway Materials (C-9, D-4), 360 pp., 2600 copies.
Standards on Glass and Glass Products (C-14), 136 pp., 1000 copies.
Standards on Petroleum Products and Lubricants (D-2), 984 pp., 7000 copies.
Standards on Bituminous Materials for Highway Constructions, Waterproofing and Roofing (D-4, D-8), 459 pp., 1700 copies.
Standards on Papers, Paper Products and Shipping Containers (D-6, D-10), 401 pp., 1500 copies.
Standards on Soaps and Other Detergents (D-12), 175 pp., 1000 copies.
Standards on Textile Materials (D-13), 788 pp., 1600 copies.
Standards on Plastics (D-120), 808 pp., 2000 copies.
Standards in Building Codes, 964 pp., 2700 copies.

Special Compilations in Prospect:

Standards on Electrical Insulating Materials (D-9), 659 pp., 1000 copies.
Standards on Rubber and Rubber-Like Materials (D-11), 760 pp., 2400 copies.

Special Publications Issued:

Summary of Proceedings of the Fifty-eighth Annual Meeting, 38 pp., 8700 copies.
1955 Gillett Lecture, "Powder Metallurgy," by V. F. Lenel, 38 pp., 2500 copies.
1955 Edgar Marburg Lecture, "A Technology for the Analysis, Design, and Use of Textile Structures as Engineering Materials," by Walter J. Hamburger, 60 pp., 2500 copies.
Symposium on Coal Sampling (STP 162), 158 pp., 2000 copies.
Symposium on Permeability of Soils (STP 163), 142 pp., 2000 copies.
Symposium on Methods of Testing Building Constructions (STP 166), 138 pp., 2000 copies.
ASTM Viscosity Indices Tables Calculated from Kinematic Viscosity (STP 168), 832 pp., 3000 copies.
Significance of Tests of Concrete and Concrete Aggregates (STP 169), 391 pp., 4000 copies.
Chemical Compositions and Rupture Strengths of Super-Strength Alloys (STP 170), 5 pp., 2000 copies.
Symposium on Basic Effects of Environment on Strength, Scaling, and Embrittlement of Metals at Elevated Temperatures (STP 171), 120 pp., 2500 copies.
Bibliographical Abstracts on Redeposition of Soil on Cotton Fabrics (STP 173), 32 pp., 2500 copies.
Symposium on Metallic Materials for Service at Temperatures Above 1600 F (STP 174), 196 pp., 3000 copies.
Bibliographical Abstracts on Evaluation of Brightening Agents for Detergent Usage (STP 177) 13 pp., 2000 copies.
Panel Discussion on Pyrometric Practices (STP 178), 48 pp., 3000 copies.
The Elevated Temperature Properties of Carbon Steels (STP 180), 67 pp., 2500 copies.
Determination of the Shear Stability of Non-Newtonian Liquids by Neal D. Lawson (STP 182), 15 pp., 2500 copies.
The Mechanical Properties of Wrought Phosphor Bronze Alloys by G. R. Gohn, J. P. Guerard and H. S. Freynik (STP 183), 118 pp., 3800 copies (1500 for Bell Laboratories and 300 for Riverside Metal Co.)
Bibliography of Fatigue References (STP 9 F), 29 pp., 500 copies.
Cumulative, Alphabetical and Grouped Numerical Index of X-ray Diffraction Data (STP 48 E), 716 pp., 1000 copies.
Bibliography on Electrical Contacts (STP 56-I), 46 pp., 1200 copies.

Special Publications in Prospect:

Report on Standard Samples and Related Materials for Spectrochemical Analysis (STP 58 C), 100 pp., 3000 copies.
Symposium on Evaluation of Insulating Oils—European Developments (STP 172), 78 pp., 2000 copies.
Symposium on Atmospheric Corrosion of Non-Ferrous Metals (STP 175), 158 pp., 3000 copies.
Symposium on Impact Testing (STP 176), 190 pp., 3000 copies.
Symposium on High Purity Water Corrosion (STP 179), 64 pp., 3000 copies.
Survey of Copper Base Alloys (DP-7)(STP 181), 250 pp., 2500 copies.
Glossary of Terms—Rubber and Rubber-Like Materials (STP 184), 128 pp., 2000 copies.
Symposium on Speed of Testing (STP 185), 134 pp., 2000 copies.
Methods for Reducing the Effect of Barometric Pressure in Measurement of Octane Number,

by Bruno R. Siegel (STP 186), 32 pp., 2000 copies.

Relaxation Properties of Steels and Super-Strength Alloys at Elevated Temperatures (STP 187), 96 pp., 2500 copies.

Symposium on Minimum Property Values of Insulating Materials (STP 188), 75 pp., 2000 copies.

Significance of Tests of Petroleum Products and Lubricants, 134 pp., 6000 copies.

ASTM Manual of Engine Test Methods for Rating Fuels, 192 pp., 2500 copies.

Selected Engineering Materials Standards of the ASTM, 476 pp., 10,000 copies.

Respectfully submitted on behalf of the committee,

R. E. HESS,
Chairman.

APPENDIX V

REPORT OF ADMINISTRATIVE COMMITTEE ON DISTRICT ACTIVITIES

During the June 1955–June 1956 period, 14 ASTM Districts sponsored 24 district meetings. In addition, five meetings were held in various cities in the Northwest part of the United States. This area may eventually be made a formal district. At these five meetings, President Fellows gave his regular district address. There was one additional meeting—a special luncheon meeting held in Toronto—which was attended by the President and Executive Secretary. The Philadelphia District Council acted as hosts for the 58th Annual Meeting, planning the ladies' entertainment, handling details of the meeting, and underwriting the entertainment program.

In connection with national meetings, the Philadelphia District is arranging for the ladies' program and the details of the dinner and entertainment for the Annual Meeting to be held in Atlantic City this year. The Northern and Southern California Districts are sponsoring the program and handling details of some of the special features for the Second Pacific Area National Meeting to be held in September of this year.

The increased enthusiasm among districts with respect to sponsorship of student membership prize awards is particularly noteworthy. The Philadelphia District acted as host for a number of the 29 recipients in that area. The Cleveland District had 14 (out of 21) students as guests at a dinner meeting and the Pittsburgh District invited the eight student membership prize recipients from the Pittsburgh area to their social evening held in honor of President Fellows. The Detroit District is also awarding eight memberships, and Southern California is sponsoring 21 awards in addition to another 28 sponsored by ASTM members in that area. Other districts are considering sponsoring this type of activity.

President Fellows presented his district address, "Research in the Electric Power Industry (Generation—Transmission—Distribution)," 15 times. Ten of the meetings were district meetings, and the other five were held in Salt Lake City, Utah; Richland, Wash.; Spokane, Wash.; Portland, Ore.; and Vancouver, B.C. New areas were host this year to the President, including New Orleans (Southwest District), Salt Lake City, and Vancouver. A number of the meetings were sponsored solely by ASTM, but the majority were cooperative undertakings between ASTM and groups such as AIEE, ASME, ASM, ACS, AIChE, and Western Society of Engineers.

President Fellows' address was devoted to the current aspects of research in the electric power industry—particularly in his own company—and included the latest developments on high-voltage aluminum cable; wood preservation—poles, cross arms, etc.; corrosion—especially galvanized steel tower transmission; tree growth control; fish fences; insulating oils; hydrocarbons—fundamental research aspects; and nuclear power.

Dr. Fritz V. Lenel, 1955 Gillett Memorial Lecturer, made a West Coast trip, speaking in Chicago, St. Louis, Los Angeles, San Francisco, and Dallas. He presented an abridged version of his lecture on "Powder Metallurgy—Now."

Several of the districts have developed their technical programs in a most interesting manner. The New England District has for the past several years held its Spring Meeting at one of the colleges or universities in the area, usually with a tour of the engineering facilities in the afternoon and a dinner meeting. The meeting this year was held at the University of Massachusetts. The New York District is following the practice of having a particular company in the area present the program, such as the meeting on January 11 entitled, "A Night With Bell Telephone Laboratories."

The Administrative Committee on District Activities again sponsored a breakfast meeting to which district council officers or their representatives were invited. These meetings have been quite helpful, serving as a means for interchange of information and ideas for promotion of the Society and its activities.

In addition to the sponsoring of technical sessions, various districts of the Society have been active in sponsoring student membership prize awards, participation in such activities as science fairs and local engineering councils, and cooperation with the Society's advisory Committee on Corrosion.

The ACDA held one meeting last year on June 28, 1955, during the Annual Meeting. Messrs. E. K. Spring, C. M. Gambrill, and H. P. Hagedorn was appointed to the committee for three-year terms. There was some discussion of the possibility of the formation of a Canadian District, and the committee went on record as favoring the establishment of a separate district in the Ontario area.

Respectfully submitted on behalf of the committee,

C. M. GAMBRILL,
Chairman.

J. S. PETTIBONE,
Secretary.

APPENDIX VI

REPORT OF THE ADMINISTRATIVE COMMITTEE ON SIMULATED SERVICE TESTING

The committee has not held a meeting yet this year, but is actively engaged in pursuing two of the activities which were of prime interest at the last ACSST meeting.

The first of these has to do with Society activities in the field of gasket materials, where it is the feeling of the members of the Administrative Committee that a much more coordinated and extensive effort should be made on gasket materials in classification (particularly on end-use basis), test methods, and specifications. The ASTM staff has been acting as liaison between the Administrative Committee and Committee D-11 in this connection, and the committee is now awaiting indications from Committee D-11 as to whether or not that group is prepared to enlarge the scope of its gasket activities to encompass the wide array of such materials included in ASTM interests.

At the request of the ACSST, Committee E-4 on Metallography has contacted all of the metals committees of the society concerning the question of the desirability of ASTM standards of macro-structures (by macro-etch testing). As of the time of this report, Committee E-4 has received answers from nearly all of the metals committees, and of the eight replies which show that the particular technical committee does have an interest in such tests, six were in favor of having such standards. Of the two which had interests but were not in favor of such standards, one indicated that the decision was based on the thought that the problem was too extensive in the numbers of structures that would have to be shown.

Another phase of ACSST interests was exemplified by the discussions pertinent to the testing interests of airframe manufacturers wherein it was disclosed that the results of high-temperature tests in which they are interested must simulate actual service requirements in that the principal consideration must be the total deformation under creep conditions rather than the minimum creep rate that is conventionally reported as the result of creep testing. Recommendations for the consideration of such data collecting are to be passed on to the Joint Committee on Effect of Temperature.

Discussion was held at the last meeting concerning the recently formed Institute of Environmental Equipment Manufacturers. The committee has kept in touch with their activities throughout the year.

Respectfully submitted on behalf of the committee,

L. L. Wyman,
Chairman.

R. E. Hess,
Secretary.

REPORT OF COMMITTEE A-1

ON

STEEL*

With all the actions on standards recommended by a large committee throughout a year's activity, it is difficult to single out those of most importance. However, it appears as though the promulgation of the Tentative Specifications for Galvanized Steel Transmission Tower Bolts (A 394 - 55 T)[1] was the highspot of the 1955–56 year. After many drafts, which attempted to resolve conflicting views, the recommendation was finally submitted to the Administrative Committee on Standards and approved for publication on September 15, 1955.

A second development which has received much attention even before publication is the proposed Tentative Recommended Practice for Selection of Steel Bar Compositions According to Section.[2] Many letters have been received from interested parties in this country and abroad requesting draft copies for use prior to publication.

Meetings.—Committee A-1 met on June 29, 1955, in Atlantic City, N. J., in conjunction with the 1955 Annual Meeting of the Society. On June 27 and 28, eighteen subcommittees, sections, and task groups convened to prepare proposals for committee action.

Approximately 250 Committee A-1 members met on January 30 and 31 and February 1, 1956, in Pittsburgh, Pa. Twenty subcommittees, sections, and task groups met on January 30 and 31 to prepare reports for the committee meeting on February 1.

Personnel.—The committee consists of 307 voting members, including 138 producers, 127 consumers, and 42 general interest members.

Nine men were appointed to special two-year terms on the Advisory Committee in recognition of their valuable service to the committee. These were C. J. Boyle, General Electric Co.; H. F. Brown, Esso Research and Engineering Co.; F. E. Foster, Riley Stoker Corp.; M. D. Helfrick, Western Electric Co.; J. E. Lattan, Taylor Forge and Pipe Works; J. G. Morrow, Steel Co. of Canada; M. A. Pinney, Pennsylvania Railroad Co.; E. J. Ruble, Assoc. of American Railroads; and A. P. Spooner, Bethlehem Steel Co.

C. B. Bryant, Chairman of Subcommittee VII on Rolled Steel Wheels and Steel Tires, has announced his retirement as Chief Engineer of the Technical Board of the Wrought Steel Wheel Industry. P. A. Archibald, Standard Steel Works Division, Baldwin-Lima-Hamilton Corp., has been appointed as the new chairman of Subcommittee VII.

F. H. Pennell, DeLaval Steam Turbine Co., has replaced T. W. Merrill, Vanadium Corp. of America, as secretary of Subcommittee VI on Steel Forgings and Billets. J. E. Lattan, Taylor Forge and Pipe Works, was appointed chairman of the Section on Forgings of Subcommittee XXII to replace F. S. G. Williams of the same company.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 18–22, 1956.

[1] 1955 Book of ASTM Standards, Part 1.

[2] The new tentative appears in the 1956 Supplement to Book of ASTM Standards, Part 1.

The committee sustained a great loss through the death of C. E. Loos, U. S. Steel Corp., on July 30, 1955. Mr. Loos had served as chairman of Subcommittee II on Structural Steel for seven years and was a vice chairman of the committee. At the 1955 Annual Meeting he was honored with an ASTM Award of Merit in recognition of his "significant leadership in improving specification requirements for structural steel." J. R. LeCron, Bethlehem Steel Co., was appointed the new chairman of Subcommittee II.

We record, with regret, the death on May 13, 1955, of C. D. Young, past chairman of the committee and past President of ASTM. Mr. Young was chairman from 1913 to 1918, and was an honorary member of the committee.

Many other well-known presently active or past members of the committee have died since the 1955 Report. These include J. W. Ames, A. B. Bagsar, H. J. French, H. H. Lester, G. A. Lilliequist, F. S. Mapes, and A. D. Morris.

A. B. Bagsar, retired Chief Metallurgical Engineer, Sun Oil Co., was an outstanding authority in the application of metals to refinery operations. Joining the Society in 1923, he made many worthwhile contributions to the work of the committee, being a member since 1933.

H. J. French, Vice-President, The International Nickel Co., has been an active participant in ASTM committee work since 1929. Mr. French had an outstanding knowledge of physical metallurgy and received many honors during his life from technical societies.

H. H. Lester, Principal Physicist, Watertown Arsenal, was an ASTM Honorary Member and a pioneer in the field on industrial radiography. Although primarily interested in nondestructive testing, Dr. Lester made important contributions to the committee.

J. W. Ames, President, W. Ames and Co., served on the committee and Subcommittee I on Steel Rails and Accessories since 1944. G. A. Lilliequist, Research Director, American Steel Foundries, was known internationally for his contributions to the field of metallurgy, being a member of the committee and Subcommittees VIII, XXIV, and XXV since 1943.

F. S. Mapes of the General Electric Co. was a testing expert, serving on Subcommittees XIII and XXVII. A. D. Morris, President, Bayonne Bolt Corp., was a well-known authority on bolting materials, serving on Subcommittees I, II, XXII and XXVI.

Election of Officers.—The officers elected for the ensuing term of two years were as follows:

Chairman, W. F. Collins.
Vice-Chairman, J. J. Kanter.
Vice-Chairman, C. L. Clark.
Secretary, H. L. Fry.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

On August 31, 1955, the Administrative Committee on Standards accepted the recommendation to revise the Tentative Specifications for Forged or Rolled Alloy Steel Pipe Flanges, Forged Fittings, and Valves and Parts for High Temperature Service (**A 182 – 55 T**). The revised specifications appear in the 1955 Book of ASTM Standards, Part 1, bearing the designation indicated above.

The Tentative Specifications for Galvanized Steel Transmission Tower Bolts (**A 394 – 55 T**) were accepted by the Administrative Committee on Standards on September 15, 1955, and appear in the 1955 Book of ASTM Standards, Part 1.

Also on September 15, 1955, the Administrative Committee accepted the recommendation to withdraw Emergency Alternate Provisions EA - A 26 and to revise Emergency Alternate Provisions **EA - A 329**. The revision of EA - A 329 appears in the 1955 Book of ASTM Standards, Part 1, as EA - A 329a.

On May 2, 1956, the Administrative Committee accepted the following recommendations:

Revision of Tentative Specifications for:

Billet-Steel Bars for Concrete Reinforcement (**A 15 - 56 T**),

Welded Steel Wire Fabric for Concrete Reinforcement (**A 185 - 56 T**),

Minimum Requirements for the Deformations of Deformed Steel Bars for Concrete, and Reinforcement (**A 305 - 56 T**).

Withdrawal of Standard Specifications for:

Welded Steel Wire Fabric for Concrete Reinforcement (**A 185 - 37**).

RECOMMENDATIONS AFFECTING STANDARDS

The committee is presenting for publication 7 new tentatives and is recommending the revision of 27 tentatives and 7 standards and the adoption as standard of 14 tentatives. In addition, one standard is recommended for reversion to tentative with revision, one standard and one tentative are being withdrawn, and seven standards are being reaffirmed.

The standards and tentatives affected, together with the revisions recommended, are given in detail in the Appendix.[3]

The recommendations in the report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Only highlights of the many current subcommittee activities will be covered in the following paragraphs:

Subcommittee II on Structural Steel is drafting a specification for high-strength steel for structural purposes. This has been requested by the American Railway Engineering Assn. Committee 15 on Iron and Steel Structure.

Subcommittee VI on Steel Forgings and Billets is continuing discussions in two special task groups on (1) hydrogen in steel forging, and (2) tests and criteria for judging the sensitivity of heavy rotor forgings to fracture. The first task group has completed the preparation of a proposed method of sampling steel forgings for hydrogen content. Members of the latter task group presented papers at an ASME session in Chicago in December, 1955. An extensive research program on brittle failure has been initiated.

With the publication of the Tentative Method for Dry Powder Magnetic Particle Inspection (E 109 - 55 T), the withdrawal of the Tentative Method of Magnetic Particle Testing and Inspection of Heavy Steel Forgings (A 272 - 44 T) is under consideration. If withdrawn, casting specifications A 27, A 148, A 216, A 217, A 351, A 353, A 356, and A 389 would be revised to refer to Method E 109 in place of Method A 272.

Two important subjects continue to be discussed in Subcommittee XI on Steel for Boilers and Pressure Vessels. These include requirements for carbon steel plates to resist graphitization at elevated temperatures and limits for residual alloys in plate steel. Also in the field of flat products, Subcommittee XIX has nearly reached agreement on a proposed specification for hot-rolled commercial quality steel sheets.

If the revision of chemical requirements for type 347 steel and the addition of type 348 steel to Specifications A 269 and A 271, as outlined in the Appendix, is approved by the Society, the Emergency Alternate Provisions EA - A 269a and EA - A 271a will no longer be needed and these will be withdrawn. Similarly, if the proposed revision of the manganese content in Specifications A 26 for Steel Tires is approved, Emergency Alternate Provisions EA – A 329a will also be withdrawn. This will eliminate the last of the Emergency Alternate Provisions requested by the committee during the last War Emergency.

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[3] See p. 112.

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee XXVII on Steel Chain along with Committee A-2 on Wrought Iron is considering the withdrawal of Specifications A 56 – 39 for Iron and Steel Chain (which falls under the joint jurisdiction of Committees A-1 and A-2). The subcommittee proposes to write a new specification covering carbon steel chain of three strength levels. Likewise Committee A-2 would write a new specification covering wrought iron chain.

This report has been submitted to letter ballot of the comemitte, which consists of 307 members; 198 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

H. B. OATLEY,
Chairman.

H. L. FRY,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee A-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

Withdrawal of Emergency Alternate Provisions for:

Heat-Treated Steel Tires (EA – A 329),
Seamless and Welded Austenitic Stainless Steel Tubing for General Service (EA – A 269) (Jointly with Committee A-10),
Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (EA – A 271) (Jointly with Committee A-10), and
Seamless and Welded Austenitic Stainless Steel Pipe (EA – A 312) (Jointly with Committee A-10).

These recommendations were accepted by the Standards Committee on November 21, 1956.

APPENDIX

PROPOSED RECOMMENDATIONS AFFECTING STANDARDS ON STEEL

In this Appendix recommendations are given affecting certain specifications, both standard and tentative, covering various steel products. The specifications appear in their present form in the 1955 Book of ASTM Standards, Part 1.

New Tentatives

The committee recommends that the following specifications and recommended practice be accepted for publication as tentative as appended hereto:[1]

Tentative Specifications for:

Chromium-Silicon Steel Valve Spring Quality Wire,

Chromium-Silicon Steel Spring Wire,

High Tensile Strength Carbon-Manganese Steel Plates for Unfired Pressure Vessels,

Factory-Made Wrought Austenitic Steel Welding Fittings,

Forged or Rolled Alloy Steel Pipe Flanges, Forged Fittings, and Valves and Parts Specially Heat Treated for High Temperature Service, and

Seamless Ferritic Alloy Steel Pipe Specially Heat Treated for High Temperature Service.

Tentative Recommended Practice for:

The Selection of Steel Bar Compositions According to Section.

Revisions of Tentatives

The committee recommends that the following 26 Specifications be revised as indicated and continued as tentative:

Tentative Specification for General Requirements for Delivery of Rolled Steel Plates, Shapes, Sheet Piling, and Bars for Structural Use (A 6 – 54 T):

Section 9 (a).—Change the limiting thickness for individual marking for steel plates from the present "$\frac{5}{16}$ in." to be "$\frac{3}{8}$ in." and add a footnote referenced after "$\frac{3}{8}$ in." to read: "For material specified for bridge construction the limiting thickness of plates to be individually identified shall be $\frac{5}{16}$ in. instead of $\frac{3}{8}$ in."

Table XXIII.—Revise to read as shown in the accompanying Table I.

TABLE I.—PERMISSIBLE VARIATIONS IN LENGTH FOR STANDARD STRUCTURAL SECTIONS.
(Revision of Table XXIII of A 6 – 54 T)

	Variations from Specified Length for Lengths Given, in.									
	To 30 ft, incl		Over 30 to 40 ft, incl		Over 40 to 50 ft, incl		Over 50 to 65 ft, incl		Over 65 ft	
	Over	Under	Over	Under	Over	Under	Over	Under	Over	Under
All standard sections......	½	¼	¾	¼	1	¼	1⅛	¼	1¼	¼

Tentative Specification for Steel for Bridges and Buildings (A 7 – 55 T):

Section 4.—Change the section title to read "Bearing Plates and Anchor Bolts" and add a new Paragraph (*c*) to read "Unless otherwise specified, all plain and threaded bars used for anchorage purposes shall be subjected to mechanical tests and shall conform to the tensile requirements of Section 7; headed bolts used for anchorage purposes, and all nuts, shall conform to the requirements of Specification A 307."

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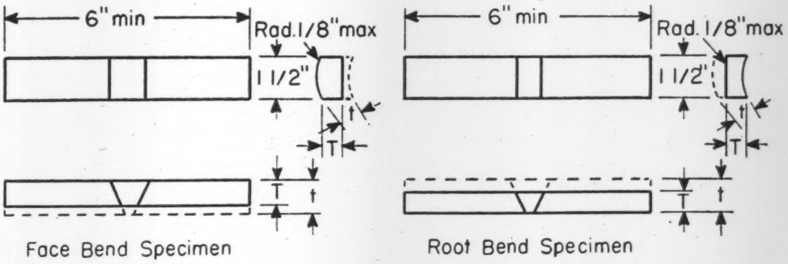
[1] The new tentatives appear in the 1956 Supplement to Book of ASTM Standards, Part 1.

Section 6.—Add a new Paragraph (*d*) to read "When tension tests are waived in accordance with Section 7 (*b*), chemistry consistent with the mechanical properties desired must be applied."

Section 7.—Change Paragraph (*b*) to read: "Shapes less than 1 sq in. in cross-section, and bars, other than flats, less than ½ in. in thickness or diameter need not be subjected to tension tests by the manufacturer."



Face Bend Specimen

Root Bend Specimen

Pipe Wall Thickness (*t*), in.	Test Specimen Thickness, in.
Up to ⅜, incl.	*t*
Over ⅜	⅜

FIG. 1.—Transverse Face- and Root-Bend Test Specimens.

Tentative Specifications for Seamless Steel Boiler Tubes (A 83 – 55 T):

Section 6.—In the table at the end of Paragraph (*a*), in the column listing outside diameter of tube, change the present "2½ and under" to read "Over ¾ to 2½ in."

Add a new sentence to Paragraph (*a*) to read "For tubing ¾ in. and less in outside diameter the width of the flange shall be not less than 15 per cent of the outside diameter of the tubing."

Tentative Specifications for Electric-Fusion-Welded Steel Pipe for High-Temperature Service (A 155 – 55 T):

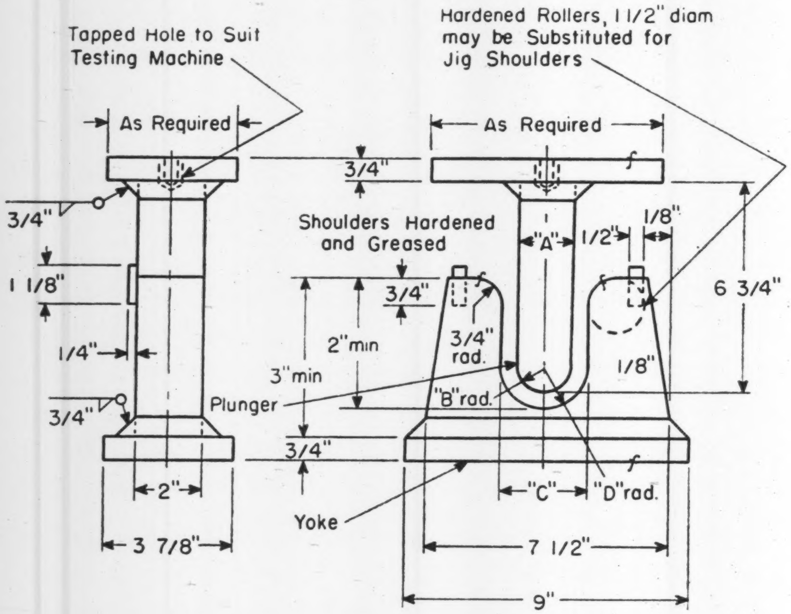
Section 8.—Change the section title to read "Transverse Guided Weld Bend Tests" and revise the text to read as follows:

8. (*a*) Two bend test specimens shall be taken transversely from the pipe. One shall be subject to a face guided bend test and the second to a root guided bend test. These specimens shall be approximately 1½-in. wide, at least 6 in. in length with the weld in the center and shall be machined in accordance with Fig. 1 (accompanying Fig. 1). One specimen shall be bent with the inside surface of the pipe against the plunger, and the other with the outside surface against the plunger. The dimensions of the plunger and the bending jig shall be in accordance with Fig. 2 (accompanying Fig. 2).

(*b*) The bend test shall be acceptable if no cracks or other defects exceeding ⅛ in. in any direction be present in the weld metal or between the weld and the pipe metal after bending. Cracks which originate along the edges of the specimen during testing, and that are less than ¼ in. measured in any direction shall not be considered.

Section 10 (*a*).—Revise the first sentence to read "One transverse tension test (Section 7 (*b*)) and two transverse guided weld bend tests (Section 8) shall be made to represent each lot of pipe."

New Figures.—Add new Figs. 1 and 2 as shown in the accompanying Figs. 1 and 2.


Tapped Hole to Suit Testing Machine
As Required
3/4"
1 1/8"
1/4"
3/4"
2"
3 7/8"
Hardened Rollers, 1 1/2" diam may be Substituted for Jig Shoulders
As Required
3/4"
Shoulders Hardened and Greased
3/4"
2" min
3" min
Plunger
3/4"
Yoke
"A"
1/2"
1/8"
6 3/4"
3/4" rad.
1/8"
"B" rad.
"C"
"D" rad.
f
7 1/2"
9"

Thickness of Specimen	*A*, in.	*B*, in.	*C*, in.	*D*, in.
3/8 in.	1½	¾	2⅜	1 3/16
t	4*t*	2*t*	6*t* + ⅛	3*t* + 1/16

FIG. 2.—Guided-Bend Test Jig.

Tentative Specifications for Electric-Resistance-Welded Steel and Open-Hearth Iron Boiler Tubes (A 178 – 55 T):

Section 7.—Revise as indicated above for Section 6 (*a*) in Specifications A 83.

Tentative Specifications for Seamless Cold-Drawn Low-Carbon Steel Heat-Exchanger and Condenser Tubes (A 179 – 55 T):

Section 7.—In Paragraph (*b*) add a new sentence to read "For tubing ¾ in. and less in outside diameter the width of the flange shall be not less than 15 per cent of the outside diameter of the tubing."

Tentative Specifications for Forged or Rolled Alloy-Steel Pipe Flanges, Forged Fittings, and Valves and Parts for High-Temperature Service (A 182 – 55 T):

Table I.—Change the chemical requirements for grade F 347 and add a new grade F 348 to read as shown in the accompanying Table II for types 347 and 348 steels.

Tentative Specifications for Alloy-Steel Bolting Materials for High-Temperature Service (A 193 – 55 T):

Section 1.—In Note 2 omit the following sentence "Upon agreement between the manufacturer and the purchaser, other grades of material may be employed, provided they otherwise conform to the requirements of these specifications."

TABLE II.—CHEMICAL REQUIREMENTS.

(Revision of Chemical Requirements in A 213, A 249, A 269, A 271, A 312, and A 376)

	Type 347	Type 348
Carbon, max, per cent	0.08	0.08
Manganese, max, per cent	2.00	2.00
Phosphorus, max, per cent	0.030	0.030
Sulfur, max, per cent	0.030	0.030
Silicon, max, per cent	1.00	1.00
Chromium, per cent	17.00 to 20.00	17.00 to 20.00
Nickel, per cent	9.00 to 13.00	9.00 to 13.00
Columbium plus tantalum, per cent	10 × C, min 1.0 max	10 × C, min 1.0 max
Tantalum, max, per cent	...	0.10

Table I.—For grades B7A and B16 change the required molybdenum contents to read "0.50 to 0.65" per cent. For grade B16 change the required carbon content to read "0.36 to 0.44" per cent and the required manganese content to read "0.45 to 0.70" per cent.

Also change the chemical requirements for grades B5, B6, B8, B8C, B8T, and B8F and add a new grade B8D covering type 348 to read as shown in the accompanying Table III.

Tentative Specifications for Carbon and Alloy Steel Nuts for Bolts for High-Pressure and High-Temperature Service (A 194 – 55 T):

Table I.—Change the chemical requirements for grades 3, 6, 8, 8C, 8T, and 8F and add a new grade 8D covering type 348 as shown in the accompanying Table III.

Sections 9 and 10.—Replace with the following new Section 9, entitled "Cone Stripping Test," renumbering subsequent sections accordingly.

9. The purpose of this test is to detect the presence of detrimental defects. The use of the conical washer described in Fig. 1 (accompanying Fig. 3) exaggerates the effect of such defects by introducing a simultaneous dilation and stripping action on the nut. The test shall consist of assembling the hardened washer and the nut on a hardened stud and applying the stripping proof load specified in Table III (accompanying Table IV) (see Fig. 2) (accompanying Fig. 4). The nuts shall be considered acceptable if the stripping load exceeds this proof load.

New Table.—Add a new Table III to read as shown in the accompanying Table IV, renumbering subsequent tables accordingly.

New Figures.—Add new Figs. 1 and 2 as shown in the accompanying Figs. 3 and 4.

Figure 1.—Delete this figure.

Tentative Specifications for Seamless Cold-Drawn Intermediate Alloy-Steel Heat-Exchanger and Condenser Tubes (A 199 – 55 T):

Section 9.—Revise Paragraph (*b*) as described above for Section 7 (*b*) in Specifications A 179.

Tentative Specifications for Seamless Alloy Steel Boiler, Superheater, and Heat-Exchanger Tubes (A 213 – 55 T):

Table I.—Add new grade T2 with the following chemical requirements:

Carbon, per cent	0.10 to 0.20
Manganese, per cent	0.30 to 0.61
Phosphorus, max, per cent	0.045
Sulfur, max, per cent	0.045
Silicon, per cent	0.10 to 0.30
Chromium, per cent	0.50 to 0.81
Molybdenum, per cent	0.44 to 0.65

TABLE III.—CHEMICAL REQUIREMENTS.
(Revision of Table I of A 193 - 55 T, A 194 - 55 T, and A 320 - 55 T)

Type	Ferritic Steels					
Identification Symbol	B-5 (A 193), 3 (A 194)		B-6 (A 193), 6 (A 194)			
Grade	5 per cent Chromium (AISI Type 501)		12 per cent Chromium (AISI Type 416)			
			With Added Sulfur		With Added Selenium	
	Range	Check Variation, Over or Under	Range	Check Variation, Over or Under	Range	Check Variation, Over or Under
Carbon, per cent	0.10 min	0.01 under	0.15 max	0.01 under	0.15 max	0.01 under
Manganese, max, per cent	1.00	0.03 over	1.25	0.04	1.25	0.04 over
Phosphorus, max, per cent	0.040	0.005 over	0.06	0.01 over	0.06	0.01 over
Sulfur, max, per cent	0.030	0.005 over	0.15 min	0.010 under	0.06	0.010 over
Silicon, max, per cent	1.00	0.05 over	1.00	0.05 over	1.00	0.05 over
Chromium, per cent	4.00 to 6.00	0.10	12.00 to 14.00	0.15	12.00 to 14.00	0.15
Molybdenum, per cent	0.40 to 0.65	0.05	0.60 max[a]	0.03 over	...	...
Selenium, per cent	...	...	...	...	0.15 min	0.03 under
Zirconium, per cent	...	...	0.60 max[a]	0.05 over	...	...

Type	Austenitic Steels					
Identification Symbol	B 8 (A 193, A 320), 8 (A 194)		B8C (A 193, A 320), 8C (A 194)		B8D (A 193, A 320), 8D (A 194)	
Grade	Unstabilized 18 Chromium-8 Nickel (AISI Type 304)		Stabilized 18 Chromium-8 Nickel (AISI Type 347)		Stabilized 18 Chromium-8 Nickel (AISI Type 348)	
	Range	Check Variation, Over or Under	Range	Check Variation, Over or Under	Range	Check Variation, Over or Under
Carbon, max, per cent	0.08	0.01 over	0.08	0.01 over	0.08	0.01 over
Manganese, max, per cent	2.00	0.04 over	2.00	0.04 over	2.00	0.04 over
Phosphorus, max, per cent	0.045	0.010 over	0.045	0.010 over	0.045	0.010 over
Sulfur, max, per cent	0.030	0.005 over	0.030	0.005 over	0.030	0.005 over
Silicon, max, per cent	1.00	0.05 over	1.00	0.05 over	1.00	0.05 over
Nickel, per cent	8.00 to 12.00	0.15	9.00 to 13.00	0.15	9.00 to 13.00	0.15
Chromium, per cent	18.00 to 20.00	0.20	17.00 to 19.00	0.20	17.00 to 19.00	0.20
Columbium plus tantalum, per cent	...	...	10 x carbon content, min	0.05 under	10 x carbon content, min	0.05 under
Tantalum, per cent	...	...	...	...	0.10 max	...

TABLE III,—(*Continued*).

Type	Austenitic Steels					
Identification Symbol	B8T (A 193, A 320), 8T (A 194)		B8F (A 193, A 320), 8F (A 194)			
Grade	Stabilized 18 Chromium-8 Nickel (AISI Type 321)		Free Machining 18 Chromium-8 Nickel (AISI Type 303)			
			With Added Sulfur		With Added Selenium	
	Range	Check Variation, Over or Under	Range	Check Variation, Over or Under	Range	Check Variation, Over or Under
Carbon, max, per cent	0.08	0.01 over	0.15	0.01 over	0.15	0.01 over
Manganese, max, per cent	2.00	0.04 over	2.00	0.04 over	2.00	0.04 over
Phosphorus, max, per cent	0.045	0.010 over	0.20	0.010 over	0.20	0.010 over
Sulfur, max, per cent	0.030	0.005 over	0.15 min	0.010 under	0.06	0.010 over
Silicon, max, per cent	1.00	0.05 over	1.00	0.05 over	1.00	0.05 over
Nickel, per cent	9.00 to 12.00	0.15	8.00 to 10.00	0.10	8.00 to 10.00	0.10
Chromium, per cent	17.00 to 19.00	0.20	17.00 to 19.00	0.20	17.00 to 19.00	0.20
Molybdenum, per cent	...	...	0.60 max[a]	0.03 over	...	...
Selenium, per cent	...	...	...	...	0.15 min	0.03 under
Titanium, per cent	5 x carbon content, min	0.05 under	...	...	...	...
Zirconium, per cent	...	...	0.60 max[a]	0.05 over	...	...

[a] At manufacturer's option; reported only when intentionally added.

Table II.—Change the chemical requirements for grade TP 347 and add a new grade TP 348 as shown in the accompanying Table II for types 347 and 348 steels.

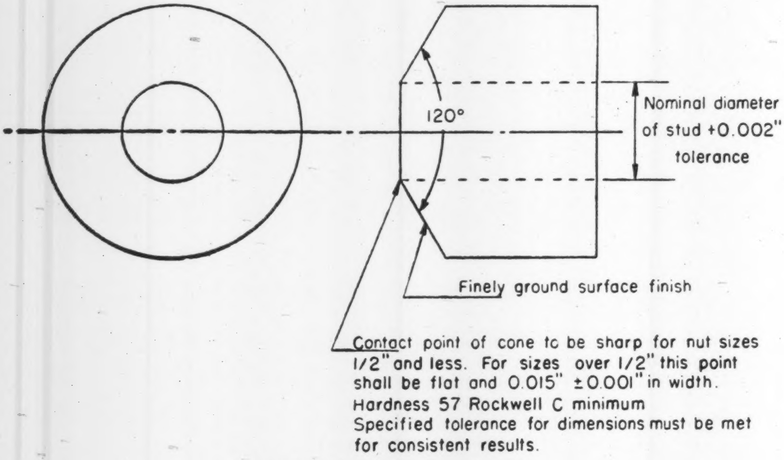

120°
Nominal diameter of stud +0.002" tolerance
Finely ground surface finish
Contact point of cone to be sharp for nut sizes 1/2" and less. For sizes over 1/2" this point shall be flat and 0.015" ±0.001" in width.
Hardness 57 Rockwell C minimum
Specified tolerance for dimensions must be met for consistent results.

FIG. 3.—Hardened Steel Cone.

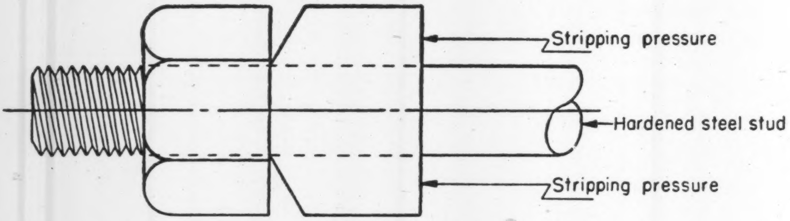

Stripping pressure
Hardened steel stud
Stripping pressure

FIG. 4.—Application of Hardened Steel Cone to Testing of Nuts.

Table III.—For ferritic steel, change the required minimum yield point from the present "25,000" to read "30,000" psi.

Tentative Specifications for Electric-Resistance-Welded Steel Heat-Exchanger and Condenser Tubes A 214 – 55 T):

Section 7.—Revise as indicated above for Section 7 (*b*) in Specifications A 179.

Tentative Specifications for Carbon-Steel Castings Suitable for Fusion Welding for High-Temperature Service (A 216 – 53 T):

Section 5 (*a*).—For grade WCB revise the carbon and manganese requirements to read as follows:

Carbon, max, per cent	0.30
Manganese, max, per cent	1.00

Section 10.—Change the first sentence in Paragraph (*b*) to read as follows, deleting the present table for hydrostatic test pressures:

> Grade WCB castings shall be individually tested after machining to the hydrostatic shell test pressures prescribed in the American Standard for Steel Pipe Flanges and Flanged Fittings (ASA No.: B 16.5—1953), for the applicable carbon steel rating for which the casting is designed and shall show no leaks.

Tentative Specifications for Electric-Resistance-Welded Steel Boiler and Superheater Tubes for High-Pressure Service (A 226 – 55 T):

Section 7.—Revise as indicated above for Section 6 (*a*) in Specifications A 83.

TABLE IV.—PROOF LOAD USING 120-DEG HARDENED STEEL CONE.[a]

(New Table III of A 194 - 55 T)

Size	Threads per Inch	Mean Thread Area, sq in.	Proof Load, lb		
			Class 1	Class 2	Class 2H, 3, 4, and 6
1/4	20	0.0318	3 800	4 400	5 150
5/16	18	0.0524	6 150	7 100	8 300
3/8	16	0.0775	8 950	10 300	12 000
7/16	14	0.1063	12 000	13 850	16 150
1/2	13	0.1419	15 700	18 100	21 100
9/16	12	0.182	19 650	22 700	26 500
5/8	11	0.226	23 900	27 550	32 150
3/4	10	0.334	33 650	38 850	45 300
7/8	9	0.462	44 300	51 100	59 650
1	8	0.606	55 150	63 650	74 250
1 1/8	8	0.790	68 000	78 500	91 600
1 1/4	8	1.000	81 250	93 750	109 350
1 3/8	8	1.234	94 250	108 750	126 850
1 1/2	8	1.492	106 700	123 100	193 600

[a] The proof load values are based on formula: conical washer proof load in pounds equals (1 − 0.30 nominal diameter of bolt) multiplied by the straight proof test load value in psi and by mean area at bolt threads. This formula cannot be used for extrapolating values for sizes beyond the range listed in this table. The straight proof test load values are:

grade 1.—130,000 psi; grade 2.—150,000 psi; grade 2H, 3, 4, and 6.—175,000 psi.

Mean thread area is based on $A_s = 3.1416 \left(\frac{E}{2} - \frac{3H}{16}\right)^2$ where E = basic pitch diameter, H = height of sharp thread. An alternate formula giving exactly the same results is $A_s = 0.7854 \left(D - \frac{0.9743}{n}\right)^2$ where D = nominal diameter of bolt, n = threads per inch.

Tentative Specifications for Factory-Made Wrought Carbon Steel and Ferritic Alloy Steel Welding Fittings (A 234 - 52a T):

Table I.—Add a new steel composition "carbon steel, grade C," identified as grade "WPC" and list as permissible raw material under pipe "A 106, grade C." Add a footnote referenced to this new grade to read "Grade WPC is a special product supplied only on agreement between the manufacturer and the purchaser."

Tentative Specifications for Welded Austenitic Stainless Steel Boiler, Superheater, Heat-Exchanger, and Condenser Tubes (A 249 - 55 T):

Table I.—Revise the chemical requirements for grade TP 347 and add a new grade TP 348 as shown in the accompanying Table II for types 347 and 348 steels.

Table II.—Add a minimum yield point of 25,000 psi for new grades TP 304 L and TP 316 L.

Section 10.—Change as indicated above for Section 7 (*b*) in Specifications A 179.

Tentative Specifications for Seamless and Welded Austenitic Stainless Steel Pipe (A 312 - 55 T):

Table I.—Revise as indicated above for Table I in Specifications A 249.

Table II.—Revise as indicated above for Table II in Specifications A 249.

Tentative Specifications for Alloy-Steel Bolting Materials for Low-Temperature Service (A 320 - 55 T):

Table I.—Revise the chemical requirements for grades B8, B8C, B8T, and B8F and add a new grade B8D covering type 348 steel as shown in the accompanying Table III. For grade L9, revise carbon and manganese requirements to read as follows:

	Range	Check Variation, Over or Under
Carbon, per cent	0.36 to 0.44	0.02
Manganese, per cent	0.65 to 0.95	0.04

Table II.—For grade L7, delete tensile requirements for sizes over $2\frac{1}{2}$ to 4 in., incl.

Tentative Specifications for Heavy-Walled Carbon and Low Alloy Steel Castings for Steam Turbines (A 356 - 52 T):

Table I.—For grade 10 change the

required chromium range from the present "2.00 to 2.50" to read "2.00 to 2.75" per cent.

Tentative Specification for 5 per cent Chromium, 0.5 per cent Molybdenum Steel Plates for Boilers and Other Pressure Vessels (A 357 – 54 T):

Section 1.—Delete the third sentence which limits the thickness to 2 in.

Table III.—Change the last listing of material thickness for bend requirements from the present "Over ⅜ to 2, incl" to read "Over ⅜."

Section 13 (a).—Add a sentence to read "Stencilling, in lieu of steel stamping, is permissible when specified."

Table IV.—Change the last listing for specified thicknesses from the present "1 to 2, incl" to read "1 and over" in.

Table VIII.—Change the last listing for specified thickness from the present "Over 1½ to 2, incl" to read "1½ to 4, excl" in. Add a new listing for specified thickness "4 to 6, excl" with permissible variations from a flat surface as follows:

Specified Width, in.	Permissible Variations from a Flat Surface, in.
To 48 incl	¼
48 excl, to 60, excl	⅜
60 to 72, excl	½
72 to 84, excl	9⁄16
84 to 96, excl	⅝
96 to 108, excl	¾
108 to 120, excl	⅞
120 to 144, excl	1
144 and over	1⅛

Tentative Specifications for Electric-Fusion-Welded Austenitic Chromium-Nickel Alloy Steel Pipe for High-Temperature Service (A 358 – 55 T):

Table I.—Add a reference to type 348 steel with a similar footnote as appears for types 309 and 310 steels.

Section 8.—Change to read as indicated above for Section 8 in Specifications A 155.

Section 10 (a).—Change to read as indicated above for Section 10 (*a*) in Specifications A 155.

New Figures.—Add new Figs. 1 and 2 as shown in the accompanying Figs. 1 and 2, respectively.

Tentative Specification for Structural Steel for Welding (A 373 – 54 T):

Section 4.—Add a new Paragraph (*e*) to read "When tension tests are waived in accordance with Section 5 (*b*), chemistry consistent with Tables I and III, and with mechanical properties desired must be applied."

Section 5.—Change Paragraph (*b*) to read as follows:

> "Shapes less than 1 sq in. in cross-section and bars other than flats less than ½ in. in thickness or diameter need not be subjected to tension tests by the manufacturer."

Tentative Specifications for Seamless Austenitic Steel Pipe for High-Temperature Central-Station Service (A 376 – 55 T):

Table I.—Change the chemical requirements for grade TP 347 and add a new grade TP 348 as shown in the accompanying Table II for types 347 and 348 steels. Add a footnote reference to grade 348 to read "This grade is intended for special purpose applications."

Tentative Specifications for Chromium-Molybdenum Steel Plates for Boilers and Other Pressure Vessels (A 387 – 55 T):

Table I.—Revise the required chro-

TABLE V.—BEND DIAMETERS.

(Revision of Table III of A 387 – 55 T)

Material Thickness, in.	Ratio of Bend Diameter to Specimen Thickness	
	Group I	Group II
1 and under	1½	2
Over 1 to 2, incl	2	2½
Over 2 to 4, incl	2	3
Over 4 to 6, incl	2½	3½

mium contents for grades A and B to read as follows:

	Grade A	Grade B
Ladle Analysis....	0.50 to 0.80	0.80 to 1.15
Check Analysis....	0.46 to 0.85	0.75 to 1.21

Table III.—Revise to read as shown in the accompanying Table V.

Section 11. –Change Paragraph (*c*) to read "On plates ¾ in. and over in thickness, tensile specimens for group II material shall be machined to the form and dimensions shown in Fig. 2 of Specifications A 20."

Delete Paragraph (*d*), relettering the present Paragraph (*e*) as new Paragraph (*d*).

Figures 1 and 2.—Delete these figures.

Section 13.—Change to read "When specified, the plates after annealing or normalizing and tempering, shall be chemically descaled or descaled by grit or sand blasting."

Tentative Specifications for Alloy Steel Castings Normalized and Drawn for High Pressure and Elevated Temperature Service (A 389 – 55 T):

Title.—Change to read "Alloy Steel Castings Specially Heat Treated for Pressure Containing Parts Suitable for High-Temperature Service."

TABLE VI.—HEAT TREATING TEMPERATURES.
(Revision of Table I of A 389 – 55 T)

Grade	Normalizing Temperature, deg Fahr	Tempering	
		Temperature, deg Fahr	Holding Time, hr
C23.......	1850 to 1950	1250 to 1350	1[a]
C24.......	1850 to 1950	1250 to 1350	12

[a] Per inch or less of thickness.

Section 1.—Revise to read as follows, deleting the present Note 3.

1. (*a*) These specifications cover alloy steel castings, which have been subjected to special heat treatment, for valves, flanges, fittings, and other pressure containing parts (Note 1) intended primarily for high-temperature service.

(*b*) The high-temperature properties of the materials covered in these specifications are dependent upon special heat treatment which is required. Although the high-temperature properties are not specified, they are implied by control of chemistry, heat treatment, and room temperature properties.

(*c*) Two grades of ferritic alloy steels are covered (Note 2). Selection will depend on the design and service conditions, mechanical properties, and high-temperature characteristics.

Section 3. –Change Paragraphs (*d*) and (*e*) to read as follows:

(*d*) *Normalizing.*—The procedure for normalizing shall consist in heating to the prescribed temperature, holding 1 hr per in. of thickness with 2 hr minimum, followed by cooling in air. If desired by the manufacturer, cooling may be in the furnace to not less than 1650 F and then in air.

(*e*) *Tempering.*—The procedure for tempering shall consist in heating to the prescribed temperature, holding for a sufficient time for the entire section to attain the required temperature, plus a minimum soaking period of the prescribed number of hours, followed by cooling in furnace or air.

Table I.—Revise to read as shown in the accompanying Table VI.

Table II.—For grade C 24, change the required chromium content from the present "0.80 to 1.20" to read "0.80 to 1.25" per cent.

TABLE VII.—WEIGHTS.
(Revision of Table II of A 391 – 55 T)

Nominal Size of Chain, in.	Maximum Weight per 100 ft, lb
¼..................	84
⅜..................	176
½..................	288
⅝..................	453
¾..................	655
⅞..................	910
1..................	1170
1⅛..................	1425
1¼..................	1765
1⅜..................	2010
1½..................	2185
1¾..................	3020

Tentative Specification for Alloy Steel Chain (A 391 – 55 T):

Section 3 (*b*).—Change the words "which are not yet specified" to read "which are not a part of this specification."

Section 7 (c).—Change the fourth sentence to read as follows by the addition of the italicized words "As soon as the beam drops or the gage halts, the machine is to be stopped *and the load reduced to the original seating load.*"

Section 9 (b).—Change the last sentence to read as follows by the addition of the italicized word "If both *additional* tests are satisfactory, the chain will be considered acceptable."

Table II.—Change the figures in the last column to read as shown in the accompanying Table VII and in the column heading change the word "nominal" to read "maximum", omitting the present Footnote *a*. Omit the word "nominal" from the title of the table.

Section 12.—Include the text of this section as a new Paragraph (*b*) to Section 10, renumbering subsequent sections accordingly.

Section 13.—Renumber as Section 12 and retitle "Workmanship and Finish." Revise to read as follows:

> 12. (*a*) The chain shall be free from injurious imperfections and shall have a workmanlike finish.
>
> (*b*) Prior to testing and inspection at the manufacturer's plant, the chain shall be free from paint or other coating which would tend to conceal defects.

Section 14.—Renumber as Section 13 and revise to read "Body chain links shall not be marked with indented characters. Other marking shall be a matter of agreement between the manufacturer and the purchaser."

Section 16.—Renumber as Section 15 and change the reference to "Section 15 (*b*)" to read "Section 14 (*b*)." Change the word "samples" to read "chains."

Section 17.—Renumber as Section 16 and change the reference to "Section 15 (*b*)" to read "Section 14 (*b*)."

REVISION OF STANDARD AND REVERSION TO TENTATIVE

The Committee recommends that the Standard Specifications for Low-Carbon Steel Track Bolts and Nuts (**A 76 - 55**) be revised as indicated and reverted to tentative status.

Section 6.—Change to read as follows:

> 6. (*a*) Tests for tensile properties shall, at the option of the manufacturer, be made on either the bars from which the bolts will be made, or on the finished bolts.
>
> (*b*) When bars from which bolts will be made are tested, they shall conform to the following full-section test requirements:
>
> Tensile strength, psi. . . . . .55 000 to 70 000
>
> Elongation in 8 in., min, per cent . . . . $\frac{1\ 500\ 000}{\text{tens. str.}}$
>
> (*c*) When finished bolts are tested, a load shall be applied between the head and nut or suitable fixture, either of which shall have sufficient thread engagement to develop the full strength of the bolt. In lieu of a bend test, a 10-deg wedge plate shall be placed under the bolt head, with the neck of the bolt aligned with the direction of uniform thickness of the wedge. When tested, in this manner, the bolt shall meet the requirements prescribed in Table I (accompanying Table VIII).

Section 7.—Change the section title to read "Bending Properties and Head Test" and revise the text to read as follows:

> 7. (*a*) The bend test specimens of full size bars from which bolts will be made shall stand

TABLE VIII.—MECHANICAL PROPERTIES FOR WEDGE TEST.

(New Table I of A 76 - 55)

Bolt Size, in,	Threads per in.	Stress Area, sq in.[a]	Breaking Load of Bolt, min, lb[b]	10-deg Wedge	
				Hole Clearance, in.	Radius on Corner of Hole, in.
½. . . .	13	0.1416	7 700	0.030	1/32
⅝. . . .	11	0.226	12 400	0.050	1/16
¾. . . .	10	0.334	18 300	0.050	1/16
⅞. . . .	9	0.461	25 300	0.063	1/16
1.	8	0.605	33 200	0.063	1/16

[a] Area at mean of root and pitch diameter of class 3 external thread.

[b] Based on a tensile strength of 55,000 psi.

being bent cold through 180 deg, flat on itself, without cracking on the outside of the bent portion.

(*b*) When testing full-size bolts for tensile properties as specified in Section 6 (*b*), the failure shall occur in the shank or the threaded portion and not at the junction of the head and shank.

Section 8.—Delete this section covering "Test Specimens," renumbering subsequent sections accordingly.

Section 9.—Renumber as Section 8 and revise to read as follows:

8. (*a*) When testing bars from which the bolts will be made, one tension test and one bend test shall be made from each heat or blow. When the heats or blows cannot be identified, one tension test and one bend test shall be made from each lot of 10 tons or fraction thereof.

(*b*) When finished bolts are tested with the 10-deg wedge plate, one tension test shall be made from each heat or blow. When the heats or blows cannot be identified, one finished bolt tension test shall be made from each lot of 10 tons or fraction thereof.

(*c*) If any test specimen shows defective machining or develops flaws, it may be discarded and another specimen substituted.

(*d*) If the percentage elongation of any tension test specimen is less than that specified in Section 6, and any part of the fracture is outside the middle third of the gage length, as indicated by the scribe scratches marked on the specimen before testing, a retest shall be allowed.

Section 11.—Renumber as Section 10 and change Paragraph (*e*) to read "A variation in the dimensions of the shoulders under the head of $\frac{1}{32}$ in. and a taper of the shoulder of $\frac{1}{32}$ in. will be permitted."

Section 13.—Renumber as Section 12 and revise to read as follows:

12. The threads of the bolts and nuts shall conform as nearly as practicable to the limits for the coarse thread series, free-fit (class 2) screws and nuts as specified in the American Standard for Track Bolts and Nuts (ASA No.: B18.10—1952).* The maximum tightness shall be 5-lb pull applied to the end of a 24-in. wrench. Before packing, nuts shall be screwed on the bolts enough turns to hold them in place until used.

ADOPTION OF TENTATIVES AS STANDARDS WITHOUT REVISION

The committee recommends that the following twelve tentative specifications and one method be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Specifications for:

Open-Hearth Carbon-Steel Rails (**A 1 – 54 T**),
General Requirements for Delivery of Rolled Steel Plates of Flange and Firebox Qualities (**A 20 – 54 T**),
Boiler and Firebox Steel for Locomotives (**A 30 – 54 T**),
Open-Hearth Iron Plates of Flange Quality (**A 129 – 54 T**),
Chromium-Manganese-Silicon (CMS) Alloy-Steel Plates for Boilers and Other Pressure Vessels (**A 202 – 54 T**),
Nickel-Steel Plates for Boilers and Other Pressure Vessels (**A 203 – 54 T**),
Molybdenum-Steel Plates for Boilers and Other Pressure Vessels (**A 204 – 54 T**),
Manganese-Vanadium Steel Plates for Boilers and Other Pressure Vessels (**A 225 – 54 T**),
High Tensile Strength Carbon-Manganese-Silicon Steel Plates for Boilers and Other Pressure Vessels (**A 299 – 54 T**),
Steel Plates for Pressure Vessels for Service at Low Temperatures (**A 300 – 54a T**),
Manganese-Molybdenum Steel Plates for Boilers and Other Pressure Vessels (**A 302 – 54 T**),
Low-Carbon High-Nickel Steel Plate for Pressure Vessels (**A 353 – 54 T**).

Tentative Method of:

Tension Testing of Steel Spring Wire (**A 318 – 48 T**).

ADOPTION OF TENTATIVE REVISION AS STANDARD

The committee recommends that the tentative revision relating to Table I of the Standard Specifications for Oil-Tempered Steel Spring Wire (**A 229 – 41**) be approved for reference to letter ballot of the Society for adoption as standard.

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* Same requirement as the A.R.E.A. Specifications for Heat Treated Carbon Steel and Alloy Steel Track Bolts and Carbon Steel Nuts.

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends that the following standards be revised immediately as indicated and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society.

Standard Specifications for Steel Tires (A 26 - 39):

Section 4.—Change the required manganese content from the present "0.50 to 0.75" to read "0.60 to 0.90" per cent.

(Note.—This change is contingent on the discontinuance of Emergency Alternate Provisions EA-A 329a, which will be submitted to the Administrative Committee on Standards.)

Standard Specification for Structural Steel for Locomotives and Cars (A 113 - 54):

Section 5.—Add a new Paragraph (*c*) to read: "When tension tests are waived in accordance with Section 6 (*b*), chemistry consistent with the mechanical properties desired must be applied."

Section 6.—Change Paragraph (*b*) to read: "Shapes less than 1 sq in. in cross-section, and bars, other than flats, less than ½ in. in thickness or diameter need not be subjected to tension tests by the manufacturer."

TABLE IX.—CHEMICAL REQUIREMENTS.
(Revision of Table I of A 131 - 55)

	Grade A		Grade B[a]		Grade C[b]	
	Ladle Analysis	Check Analysis	Ladle Analysis	Check Analysis	Ladle Analysis	Check Analysis
Carbon, max, per cent	...	...	0.21	0.25	0.24	0.28
Manganese, per cent	...	...	0.80 to 1.10	0.76 to 1.14	0.60 to 0.90	0.56 to 0.94
Phosphorus, max, per cent	0.04	0.05	0.04	0.05	0.04	0.05
Sulfur, max, per cent	0.05	0.063	0.05	0.063	0.05	0.063
Silicon, per cent	...	...	...	...	0.15 to 0.30	0.13 to 0.33

[a] When cold flanging material is specified, the manganese range for ladle analysis may be reduced to 0.60 to 0.90 per cent and for check analysis to 0.56 to 0.94 per cent.

[b] Plate steels produced to the requirements of grade C shall be made with fine grain practice.

Standard Specification for Structural Steel for Ships (A 131 - 55):

Section 1.—Add a new sentence to read: "The specification is limited to material up to 2 in., incl, in thickness."

Table I.—Change to read as shown in the accompanying Table IX.

Section 4.—In Paragraph (*b*) add the following immediately after the first sentence. "Material conforming to the requirements of class C will also be accepted."

Add a new Paragraph (*d*) to read: "When tension tests are waived in accordance with Section 5 (*b*), chemistry consistent with the mechanical properties must be applied."

Section 5.—Change Paragraph (*b*) as indicated above for Section 6 (*b*) in Specification A 113.

Standard Specifications for Heat-Treated Carbon-Steel Bolting Material (A 261 - 54):

Table I.—Delete the minimum silicon requirement.

Standard Specifications for Seamless and Welded Austenitic Stainless Steel Tubing for General Service (A 269 - 55):

Table I.—Change the chemical requirements for grade TP 347 and add a new grade TP 348 to read as shown in the accompanying Table II for types 347 and 348 steels.

Standard Specifications for Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (A 271 – 55):

Table I.—Change as described above for Table I in Specifications A 269.

ADOPTION OF TENTATIVE AS STANDARD WITH REVISION

The committee recommends that the Tentative Specifications for Nickel-Chromium-Molybdenum Steel Bars for Springs (**A 332 – 51 T**) be approved for reference to letter ballot of the Society for adoption as standard with revisions as follows:

Table I.—For grade 8650H change the carbon range from the present "0.46 to 0.54" to read "0.47 to 0.54" per cent.

Table II.—For grade 8650H change the minimum hardness value at the $\frac{1}{16}$-in. position from the present "58" to read "59" Rockwell C.

WITHDRAWAL OF SPECIFICATIONS

The committee recommends that the Tentative Specification for Chromium-Molybdenum Steel Plates for Boilers and Other Pressure Vessels (**A 301 – 54a T**) be withdrawn. The grades of steel covered by this specification are now included in the Tentative Specification for Chromium-Molybdenum Steel Plates for Boilers and Other Pressure Vessels (**A 387 – 55 T**).

It is also recommended that the Standard Specifications for Carbon-Steel Castings for Valves, Flanges, and Fittings for High-Temperature Service (**A 95 – 44**) be withdrawn since it appears as though most, if not all, of this material is now purchased in accordance with the Tentative Specifications A 216.

REPORT OF COMMITTEE A-2
ON
WROUGHT IRON*

Committee A-2 held one meeting during the year, in Atlantic City, N. J., on June 28, 1955.

A. D. Morris, chairman of the committee since its reactivation in 1951, died on October 27, 1955. Vice-Chairman D. M. Stembel has been acting as chairman since that time.

The committee consists of 31 members, of whom 5 are classified as producers, 20 as consumers, and 6 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, L. S. Crane.
Vice-Chairman, W. H. Paul.
Secretary, O. M. Tishlarich.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Tubes and Pipe (O. M. Tishlarich, chairman) is revising the maximum manganese content of Standard Specifications for Welded Wrought Iron Pipe (**A 72 - 55**) in order to bring it in line with the other specifications for wrought iron.

The Tentative Specifications for Cold-Drawn Wrought Iron Heat-Exchanger and Condenser Tubes (**A 382 - 55 T**) were submitted to the ASME Boiler and Pressure Vessel Committee for consideration as a suitable material specification in Section II of the Boiler and Pressure Vessel Code.

Subcommittee IV on Chain Iron and Iron Chain (W. H. Paul, chairman) is revising the Standard Specification for Iron and Steel Chain (**A 56 - 39**) in conjunction with Subcommittee XXVII on Steel Chain of Committee A-1. It was decided to separate the two chain materials (iron and steel) by writing a separate specification for each material.

The revised specification for iron chain, entitled "Proposed Tentative Specifications for Wrought Iron Crane Chain" and retaining the A 56 designation, was submitted to May 23, 1956, to the entire membership of Committee A-2 for comments. A revised draft, incorporating the comments received, will be submitted to the committee for letter ballot subsequent to the Annual Meeting.

Respectfully submitted on behalf of the committee,

D. M. STEMBEL,
Vice-Chairman.

L. S. CRANE,
Secretary.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 18–22, 1956.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee A-2 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision and Reversion to Tentative of:

Standard Specifications for Wrought Iron Pipe (A 72 - 55), and
Standard Specifications for Iron and Steel Chain (A 56 - 39), with a change in title to read "Specifications for Wrought Iron Crane Chain."

The revision of Specifications A 72 was accepted by the Standards Committee on September 5, 1956, and the revision of Specifications A 56 was accepted on November 14, 1956; the revised specifications appear in the 1956 Supplement to Book of ASTM Standards, Part 1.

REPORT OF COMMITTEE A-3

ON

CAST IRON*

Realizing the importance to the American cast-iron industry of international standardization, late in 1955 Committee A-3 recommended to the American Standards Association that America participate in the work of ISO Committee 25 on Cast Iron. Following clearance of this participation, the Advisory Subcommittee of Committee A-3, with the addition of several representatives of Committee A-7 on Malleable Iron, was set up as the group responsible for American participation. Two ASTM members represented the United States at the September 28, 29, and 30, 1955, meetings of ISO 25 in London—H. W. Stuart, U. S. Pipe and Foundry Co., and H. W. Lownie, Jr., Battelle Memorial Inst. Subsequently the following appointments were made on task forces of ISO 25:

Task Force on Malleable Iron—W. A. Kennedy, Grinnell Co.

Task Force on Nodular Iron—T. E. Eagan, Cooper-Bessemer Corp.

Task Force on Methods of Testing—G. A. Timmons, Climax Molybdenum Co.

There were two meetings of Committee A-3 during the year: in Atlantic City, N. J., on July 1, 1955, and in Buffalo, N. Y., on March 1, 1956. Many subcommittee meetings were held in conjunction with the main committee.

At the July 1, 1955, meeting, it was reported that the elevated temperature tests on cast iron at the Southern Research Inst. (under the guidance of the ASTM-ASME Joint Committee on Effect of Temperature on the Properties of Metals) have been continued. The test temperature had been lowered from 1000 F to 800 F. One of the alloys had been tested for 2000 hr and was carrying one-half of its former room temperature strength successfully.

The committee revised its by-laws during the year to provide for an assistant secretary, with his duties to be assigned by the incumbent secretary.

The officers elected for the ensuing term of two years are as follows:

Chairman, D. E. Kause.

Vice-Chairman, T. Eagan.

Secretary, R. A. Clark.

Assistant Secretary, H. W. Lownie, Jr.

The committee consists of 124 members, of whom 42 are classified as producers, 50 as consumers, and 32 as general interest members.

A new subcommittee on austenitic cast iron has been organized with Hugo Larson, American Brake Shoe Co., as chairman.

Subcommittee XXIII on Research (J. S. Vanick, chairman) has been disbanded since the research work for which it was organized is now being carried on by the ASTM-ASME Joint Committee on Effect of Temperature on the Properties of Metals. Subcommittee XV on Impact Testing (J. T. MacKenzie, chairman) also has been disbanded. This work will be carried on in Subcommittee XI on Methods of Testing.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

On September 15, 1955, the Administrative Committee on Standards accepted the new Tentative Specifications for Cast Nodular Iron for Pressure-Containing Parts for Use at Elevated Temperatures (A 395 - 55 T) and for High-Strength Nodular Iron Castings (A 396 - 55 T). These specifications are available in separate form.

REVISION OF TENTATIVE

The committee recommends that the Tentative Specifications for Cast Nodular Iron for Pressure Containing Parts for Use at Elevated Temperatures (**A 395 - 55 T**)[1] be revised as follows and continued as tentative:

Section 9 (*b*).—Revise Item (*3*) to read as follows:

> (*3*) The plugs, where practical, shall conform in all dimensions to the standard I.P.S. pipe plugs. In addition, they shall have full thread engagement corresponding to the thickness of the repaired section. Where a tapered plug is impractical because of the excess wall thickness in terms of plug diameter and coincident thread engagement, other types of plugs may be used provided both full engagement and effective sealing against pressure are obtained. Where possible the ends of the plugs should be ground smooth after installation to conform to the inside and outside contours of the wall of the pressure vessel or pressure part.

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions of the following two standards as indicated, and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Specifications for Gray Iron Castings (A 48 - 48):[2]

Table I.—Add a new class 45, to have a minimum tensile strength of 45,000 psi.

Table II.—Add a new class 45, with the following transverse test requirements:

Diameter of Test Bar, in.	Transverse Breaking Load, min. lb
0.875	1540
1.2	2800
2.0	9700

Section 6.—Add a sentence to Paragraph (*a*) to read: "When neither the manufacturer nor the purchaser have taken steps to agree upon the controlling section of the casting, the size of test bar to be cast shall be determined by the manufacturer."

Revise Paragraph (*b*) to read as follows:

> (*b*) The correlation in Paragraph (*a*) is only approximate and may need modification for complicated castings with sections thinner than 0.50 in. The test bar size, type, and number may then be determined by agreement between the manufacturer and the purchaser.

Section 12.—Change the word "physical" to read "mechanical."

Note 1.—Add new class 45 to the listing of high-strength irons.

Standard Specifications for Gray Iron Castings for Pressure-Containing Parts for Temperatures Up to 650 F (A 278 - 53):[2]

Section 4.—In Paragraph (*b*) change the words "less than 1½ in. in thickness" to read "less than 2 in. in thickness."

In Paragraph (*c*) change the words "greater than 1½ in. in thickness" to read " 2 in. or more in thickness."

Add a new Paragraph (*e*) to read as follows:

> (*e*) For castings having a controlling section greater than 2 in., where it is impractical to establish a satisfactory degree of correlation between test bars and castings, the castings shall be considered as having met the tensile strength of the class specified when test coupons cut from the castings or integral projections thereof, the cross-section of which is not less than that of the controlling section of the casting, have a

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[1] Available as a separate reprint.

[2] 1955 Book of ASTM Standards, Part 1.

tensile strength not less than 80 per cent of the specified class.

Section 10.—Change Paragraph (*b*) to read as follows:

> (*b*) Surface imperfections or minor defects not exceeding 20 per cent of the thickness of the section may be repaired by plugging provided the diameter of the plug does not exceed its thickness. Seepage around chaplets, and minor leakage defects may be repaired by plugging, as directed above provided a radiographic inspection over the extent of the defect shows that the surrounding metal is sound. Castings showing a major leak under the specified pressure test shall be rejected.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Several completed activities have already been covered in this report. The following is intended only to explain certain relatively important current subcommittee activities.

Subcommittee I on Pig Iron (H. W. Stuart, chairman) is discussing the possibility of including in Specifications A 43 grades of pig iron particularly suitable for production of nodular iron.

Subcommittee XI on Methods of Testing (G. A. Timmons, chairman) is joining with Subcommittee VI on General Castings (H. W. Lownie, Jr., chairman) in establishing a task group to review the design of test bars and the testing of bars for determination of tensile strength of gray iron.

Subcommittee XXVI on Low Temperature Properties of Cast Iron (George Dinges, chairman) has reviewed several preliminary drafts of a recommended practice for the application of cast irons to low temperature services and is preparing a new draft which will incorporate the suggestions offered as a result of the previous reviews.

This report has been submitted to letter ballot of the committee, which consists of 124 voting members; 91 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

H. W. STUART,
Chairman.

T. E. EAGAN,
Secretary.

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE A-5

ON

CORROSION OF IRON AND STEEL*

Committee A-5 on Corrosion of Iron and Steel held two meetings during the year: on June 28, 1955 at Atlantic City, N. J., and on February 28, 1956, at Buffalo, N. Y.

The committee lost through retirement First Vice-Chairman J. G. Thompson, of the National Bureau of Standards. H. F. Hormann of the Consolidated Edison Co. of New York was elected to succeed Mr. Thompson for the unexpired term.

The committee consists of 110 members, of whom 41 are classified as producers, 36 as consumers, and 33 as general interest members.

The officers elected for the ensuing term of two years were as follows:

Chairman, Marc Darrin.
First Vice-Chairman, H. F. Hormann.
Second Vice-Chairman, E. F. Lundeen.
Secretary, C. P. Larrabee.

The committee has requested the Society to publish a compilation of all the standards under the jurisdiction of Committee A-5 together with Specification for Slab Zinc (B 6), and Specifications for ACSR Core Wire (B 245 and B 261).

NEW TENTATIVE

The committee recommends that the Specification for Zinc-Coated Steel Poultry Netting be accepted for publication as tentative as appended hereto.[1] This specification covers an item not heretofore covered by ASTM specifications.

This recommendation has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee VII on Methods of Test (O. B. Ellis, chairman) continues its investigation of non-destructive methods, such as use of magnetic gages for determining thickness of metallic coatings.

Subcommittee XI on Sheet Specifications (E. F. Lundeen, chairman) is reviewing the Specification for Galvanized Iron or Steel Sheets, Coils, and Cut Lengths (A 93 - 54 T) with a view to adapting this specification to the two major processes now used for producing galvanized sheets. Specification for Long Terne Iron or Steel Sheets, Coils, and Cut Lengths (A 308 - 53 T) is being reviewed to include sampling methods for weight of coating tests on narrow strips and a minimum check limit by the single spot test.

Subcommittee XII on Wire Specifications (J. F. Occasione, chairman) prepared the Tentative Specification for Zinc-Coated Steel Poultry Netting appended hereto.[1] A proposed Specification for Zinc-Coated Armor Wire has been drafted. The subcommittee made plans to prepare a specification covering flat armoring tape or wire. The subcommittee is reviewing the Specifications for Chain Link Fence Fabric (A 392 - 55 T) giving particular attention to the reinstatement and use of the Preece test. Specifications A 116 and A 121 are being revised to clarify the sampling clauses.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 18–22, 1956.

[1] The new tentative appears in the 1956 Supplement to Book of ASTM Standards Part 1.

[2] The letter ballot vote on this recommendation was favorable; the results of the vote are on record at the ASTM Headquarters.

Subcommittee XIII on Hardware Specifications (B. J. Barmack, chairman) reviewed the specifications under its jurisdiction and recommended various editorial revisions in A 123 - 53, A 143 - 46 and A 153 - 53. The results of over one thousand tests of coating weights on hardware will be studied with the objective of revising the present weights of coating in Table I of Specifications A 153. New projects include preparation of a recommended practice on inspection of galvanizing and study of the problem of regalvanizing of material by job galvanizers.

Subcommittee XIV Sheet Tests (C. P. Larrabee, chairman) inspected the corrugated sheets under atmospheric exposure test at State College, Pa., on April 27, 1956. The data from this and previous inspections are summarized in Table I. The one sheet remaining at Altoona, Pa., is still unperforated.

TABLE I.—LIFE OF CORRUGATED GALVANIZED AND BLACK SHEETS, 26 BY 30 IN.
Exposed at State College, Pa., on April 23, 1926.

Sheet	No. 16 Gage, 2.5 oz per sq ft[a]		No. 22 Gage, 2.5 oz per sq ft[a]			No. 22 Gage, 2.0 oz per sq ft[a]			No. 22 Gage, 1.5 oz per sq ft[a]			No. 22 Gage, 1.25 oz per sq ft[a]			No. 22 Gage, 0.75 oz per sq ft[a]			No. 28 Gage, 0.75 oz per sq ft[a]			No. 22 Gage, Uncoated
	Years To		Years To			Years To			Years To			Years To			Years To			Years To			
	1st Rust	100 per cent Rust	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	1st Rust	100 Per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	Years To Perforation
R (T)	24.0	*	26.0	*	*	19.0	*	*	17.5	*	*	14.5	*	*	c	....	....	c	....	....	26.0[b]
(B)	27.0	*	29.0	*	*	20.0	*	*	16.5	*	*	15.0	*	*	c			c			
V (T)	27.0	*	21.5	*	*	18.5	*	*	16.0	*	*	15.0	*	*	9.5	23.0	*	c			24.0(V)
(or U) (B)	24.0	*	29.0	*	*	25.0	*	*	15.5	*	*	16.5	*	*	10.5	24.0	*	c	....	....	25.0(U)[b]
N (T)	c	....	20.5	*	*	22.5	*	*	17.5	*	*	12.0	*	*	6.5	20.5	*	11.0	23.0	*	28.0
(B)	c		28.0	*	*	22.5	*	*	15.5	*	*	13.5	*	*	9.0	23.0	*	11.5	*	30.0	
T (T)	27.0	*	*	*	*	21.0	*	*	16.0	*	*	15.0	*	*	9.0	26.0	*	9.0	24.0	*	*
(B)	*	*	*	*	*	23.0	*	*	14.5	*	*	13.5	*	*	10.5	22.5	*	12.0	25.0	*	
Y (T)	21.5	*	22.5	*	....	19.5	*	....	17.5	*	....	14.5	*	....	10.0	22.5	....	9.5	24.0	....	25.0
(B)	22.5	*	29.0	*	....	19.0	*	....	18.5	*	....	14.5	29.0	....	10.5	30.0	....	13.0	26.0	....	
S (T)	22.0	*	28.0	*	*	25.0	*	*	19.0	*	*	14.5	*	*	10.5	21.5	*	11.5	22.0	*	*
(B)	22.5	*	*	*	*	28.0	*	*	19.5	*	*	15.5	30.0	*	11.0	21.5	*	11.5	21.5	*	
C (T)	c	....	22.5	*	*	17.5	*	*	16.0	*	*	14.5	*	*	9.5	24.0	*	c	....	....	*
(B)	c		*	*	*	21.5	*	*	18.5	*	*	14.5	30.0	*	10.0	24.0	*	c			
K (T)	23.0	*	25.0	*	*	22.5	*	*	17.5	*	*	14.5	*	*	11.5	24.0	*	10.5	23.0	*	*
(B)	24.0	*	30.0	*	*	28.0	*	*	21.0	*	*	16.5	*	*	11.5	22.5	*	11.5	21.0	*	
W (T)	....	....	29.0	*	*	25.0	*	*	16.5	*	*	13.5	*	*	c	....	....	c	....	....	27.0
(B)			*	*	*	28.0	*	*	15.0	*	*	14.5	30.0	*	c			c			
Average	....	....		....	....	22.5	....	....	17.0	....	....	14.6			10.0	23.5		11.1	24.0		

* Data not yet available.
[a] Coating weight is "oz per sq ft" of sheet (both sides).
[b] Inadvertently omitted from 1952 report.
[c] There were no specimens exposed.

The subcommittee is studying methods of test for aluminum-coated and zinc-coated steels and plans to initiate exposure tests when a sufficient number of producers are manufacturing aluminum-coated steel.

The subcommittee is cooperating with the American Welding Society in its inspection of metallized coatings exposed at ASTM test sites. These steel panels sprayed with either zinc or aluminum were exposed during 1953 and 1954 at seven atmospheric sites, submerged in sea water, and also exposed at the mean-tide level. A total of 1164 zinc-coated specimens and an equal number of aluminum-coated specimens were included in the tests. The aluminum-coated specimens were exposed in three conditions: as sprayed, as sprayed plus primer and one coat of aluminum vinyl paint, and as sprayed plus primer and two coats of aluminum vinyl paint. The zinc specimens were similarly exposed, except that two coats of clear chlorinated rubber paint were substituted for the primer and two coats of aluminum vinyl paint. A total of 624 sprayed panels were submerged and 672 panels were exposed at mean tide. Results of one- and two-year inspections of the atmospheric specimens showed very little, if any, change. The final merits of individual systems will be determined by the number of years of exposure before appearance of rust on the base metal. No rust has been reported in the atmospheric tests.

Subcommittee XV on Wire Tests (F. M. Reinhart, chairman) made annual inspections of wires exposed at various ASTM test sites and a report of inspection data is appended hereto.[2]

Subcommittee XVI on Hardware Tests (A. Mendizza, chairman) announced that the hardware-test installation at Key West, Fla., had been destroyed and all samples had been lost. The subcommittee is proceeding with a new hardware test, and samples will be exposed on the ASTM test sites in Newark, N. J., and Kure Beach, N. C.

This report has been submitted to letter ballot of the committee, which consists of 110 members; 99 members returned their ballots, of whom 93 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

A. P. JAHN,
Chairman.

C. P. LARRABEE,
Secretary.

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[2] See p. 133.

REPORT OF SUBCOMMITTEE XV ON ATMOSPHERIC EXPOSURE TESTS OF WIRE AND WIRE PRODUCTS

(COVERING 1955 INSPECTIONS)

This report summarizes the results of the atmospheric corrosion tests on wire and wire products through 1955. Detailed results are reported in the odd-numbered years, and reference to these earlier reports of Committee A-5 on Corrosion of Iron and Steel, which appeared in the Society's *Proceedings* for 1939, 1941, 1943, 1945, 1947, 1949, 1951, 1953, and 1955 will be helpful in studies of these data. These earlier reports contain descriptions of the test methods, micrographs, and other characterizations of specimens in the test as well as the data assembled during previous inspections.

The materials being tested are mostly bare and zinc-coated steel wires and wire products, but copper-covered and lead-coated steel wires and chromium and chromium-nickel steel wires are also included. The coatings are expressed in weight terms of ounces per square foot of surface. For comparisons of coating thickness, 1 oz per sq ft of surface may be considered as averaging 0.0017 in. thick for zinc coatings, 0.0013 in. thick for copper coatings, and 0.0010 in. thick for lead coatings.[1]

Specimens of wires and wire products were exposed for approximately 19 yr at eleven locations. The location of these sites and their general classification regarding atmospheres are as follows:

Test Location	General Type of Atmosphere	Remarks
Pittsburgh, Pa. (abandoned 1951)	Severe industrial	On Brunot Island in the Ohio River about 2 miles west of the city
Bridgeport, Conn.	Industrial	In the city
Sandy Hook, N. J. (abandoned 1952)	Seacoast	About 300 yards from the Atlantic Ocean
Santa Cruz, Calif.	Rural (marine)	About 3 miles from the Pacific Ocean
State College, Pa.	Rural	Central Pennsylvania
Lafayette, Ind.	Rural	Wabash River Valley
Ames, Iowa	Rural	Central Iowa
Manhattan, Kans.	Rural	Kansas River Valley
Ithaca, N. Y.	Rural	Central New York
College Station, Texas	Rural	About 120 miles inland
Davis, Calif.	Rural	About 80 miles inland

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[1] The lead-coated wires in these tests were lead coated over a bonding coat of zinc. See *Proceedings*, Am. Soc. Testing Mats., Vol. 43, p. 87 (1943) for analysis of lead coatings.

TABLE A.—REPORT OF GROUP AVERAGES THROUGH 1955—19 YEARS OF EXPOSURE.

Abbreviations and symbols used to designate appearance or state of corrosion: M = metallic; G = gray; MG = intermediate between "metallic" and "gray"; Y = yellowed or rust stained but not showing actual rough rust of base metal; R = rough rust of base metal, figures preceding R (as 5, 10, 50, 100) are percentages; PHR, PHY = rusted or yellowed in pinholes; SR, SY = speckled appearance of rust or yellowing; Br, Gn, respectively, signify corroded to brown or green appearance; D = dark or dirt or soot excluding a better observation; GY = predominantly gray but showing indication of Y. Under each location the single center column designates the state of corrosion for those groups, which as a group, have not reached 1R or 100R.

Coating Type or Weight of Zinc, oz per sq ft of surface	Number in Group	Bridgeport, Conn.		State College, Pa.		Lafayette, Ind.		Ithaca, N. Y.		Ames, Iowa		College Station, Tex.		Manhattan, Kans.		Santa Cruz, Calif.		Davis, Calif.	
		Average of Observed Years of Exposure until 1R and to 100R or State of Corrosion after Approximately 19-yr Exposure																	
		1R	100R	1R	100R	1R	100R	1R	100R	1R	100R	1R	100R	1R	100R	1R	100R	1R	100R
*UNFABRICATED WIRES																			
0.20 to 0.30	6	2.1	2.8	5.1	6.2	4.6	5.6	5.6	6.2	6.3	7.5	8.5	12.8	10.4	93R	12.6	17.0	12.3	55R
0.25 to 0.35	8	2.3	3.5	4.9	6.7	4.8	6.3	5.6	7.3	6.9	9.3	9.0	91R	10.3	73R	12.3	75R	41R	
0.35 to 0.45	7	2.9	3.7	6.6	8.4	5.5	6.8	6.7	8.3	8.4	11.4	11.5	94R	9R		12.8	23R	GY	
0.45 to 0.55	10	3.8	5.6	7.9	10.3	7.1	9.2	8.3	10.4	10.7	99R	12.8	9R	GY		13.0	GY	GY	
0.50 to 0.60	5	4.7	5.7	9.5	11.7	8.5	10.0	9.3	11.2	12.1	99R	1R		GY		GY		G	
0.60 to 0.70	12	5.4	7.1	10.2	13.2	8.8	11.4	10.4	13.0	66R		3R		GY		GY		G	
0.70 to 0.85	10	5.7	7.1	11.3	14.3	9.8	11.9	11.1	13.3	64R		2R		GY		GY		G	
0.80 to 1.00	11	6.8	8.9	90R		11.2	14.4	13.1	99R	18R		GY		GY		G		G	
1.20 to 1.35	2	9.0	11.5	25R		16.5	75R	43R		4R		G		MG		...		...	
1.60 to 1.80	11	11.8	99R	1R		1R		2R		G		G		MG		MG		MG	
2.70 to 3.00	3	GY		G		G		G		G		G		MG		MG		MG	
Cu Coat	2	Gn		Gn		Gn		Gn		Br		Br		Br		Br		Br	
Pb Coat	4	Y		60PHR+Y		22R		27R+Y		44R		7R+21Y		10R+PHY		37SR		31SR	
12–14% Cr	6	GY		G		5R		Y		SY		4R+15Y		M		5R+65SR		7R+38SR	
18%Cr–8% Ni	3	G		MG		M		M		SY		MG+Y		M		MG		MG	

					WIRE STRAND					
0.69 to 0.93	4	9.9 15.3	GY	15.0 22R	17R	G	G	MG	G	G
1.03 to 1.17	3	16.5 68R	GY	7R	GY	G	G	MG	MG	MG
1.32 to 1.51	4	75R	GY	2R	GY	G	G	MG	MG	MG
1.71 to 2.47	1	GY	G	G	G	G	G	MG	MG	MG
Pb Coat	1	Y	PHR	40PHR	20R	63R	10PHR	4R	68SR	10R
					CHAIN LINK FENCE					
0.29 to 0.52[a]	3	2.9 4.8	6.4 9.1	5.7 9.0	5.9 11.5	No Specimens Exposed	No Specimens Exposed	No Specimens Exposed	18.8 43R	18.8 25R
1.13 to 1.22[a]	2	8.9 12.0	14.5 60R	14.0 90R	18.9 50R				MG	MG
2.09 to 2.92[a]	2	3R	1R	G	G				MG	MG
1.40 to 1.97[b]	9	9.9 13.0	6R	15.5 46R	26R				MG	MG
4.21[b]	1	Y	GY	GY	GY				MG	MG
Cu Coat	1	Gn	DGn	Gn	Gn				Br	Br
Pb Coat	1	Y	PHR	50PHR	13R				35SR	35SR
18% Cr–8% Ni	1	MG	M	MG	M				MG	MG

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[a] Coated before weaving.

[b] Coated after weaving.

* Farm field fence and barbed wire made from these same wire lots corrode at about the same rate as the unfabricated wire except that in the case of farm field fence the bottom part of the fence corrodes less than the upper part; in the case of barbed wire the barbs show rust a year or two before rust appears on the main wire.

TABLE B.—REPORT OF GROUP AVERAGES THROUGH 1955—19 YEARS OF EXPOSURE.

Abbreviations and symbols used to designate appearance or state of corrosion: M = metallic; G = gray; MG = intermediate between "metallic" and "gray"; Y = yellowed or rust stained but not showing actual rough rust of base metal; R = rust of base metal, figures preceding R (5, 10, 50, 100) are percentages; PHR, PHY = rusted or yellowed in pinholes; SR, SY = speckled appearance of rust or yellowing; Br, Gn, respectively, signify corroded to brown or green appearance; D = dark or dirt or soot excluding a better observation; GY = predominantly gray but showing indication of Y; Brb = barbs.

Coating Group or Weight of Zinc, oz per sq ft of surface	Number in Group	State College, Pa. (17.4 yr)	Lafayette, Ind.	Ames, Iowa	Manhattan, Kans.	Ithaca, N. Y.	Santa Cruz, Calif.	College Station, Tex.	Davis, Calif.
FARM FIELD FENCE									
0.20 to 0.30	6	100R	100R	100R	68R	100R	92R	100R	78R
0.25 to 0.35	5	100R	100R	100R	44R	100R	79R	89R	51R
0.35 to 0.45	6	100R	100R	100R	19R	100R	49R	73R	18R
0.45 to 0.55	6	100R	100R	100R	3R	100R	28R	14R	GY
0.50 to 0.60	7	98R	100R	89R	GY	100R	9R	4R	GY
0.60 to 0.70	8	90R	97R	64R	GY	99R	11R	4R	GY
0.70 to 0.85	8	84R	98R	62R	MG	99R	G	GY	G
0.80 to 1.00	8	61R	89R	3R	G	80R	5R	1R	MG
1.60 to 1.80	3	GY	GY	G	MG	G	MG	G	MG
2.76	2	G	G	G	MG	G	MG	G	MG
12–14% Cr, cold drawn	1	G	SY	SY	MG	G	10R–85SR	4R	80SR
12–14% Cr, air quenched	1	MG	MG	SY	M	G	5R–40SR	2R	40SR
18% Cr–8% Ni	1	MG	M	M	M	M	MG	MG	MG
Copper covered (7.42 oz per sq ft)	1	Gn	Gn	Br	DBr	Gn	Gn	Br	Br
Lead coated (1.48 oz per sq ft)	1	100PHR	10PHR	30R	10R	25R	50SR	15R	40SR
BARBED WIRE									
0.20 to 0.30	3	100R	100R	100R	73R	100R	83R	80R	52R
0.35 to 0.45	3	100R	100R	100R	32R	100R	28R	15R	1R
0.50 to 0.60	3	100R	100R	58R	3R	95R	15R	5R	GY
0.70 to 0.85	3	92R	100R	25R	MG	100R	G	GY	MG
0.80 to 1.00	2	13R	55R	G—Brb15R	MG	14R	G	G	MG
1.60 to 1.80	3	11R	20R	G—Brb33R	MG	46R	G	G	MG
Heavy Coating	3	G—Brb30R	GY—Brb100R	G—Brb33R	MG	12R	G	G	MG
Copper covered (3.97 oz per sq ft)	2	Gn	Gn	Br	DBr	Gn	Gn, Brb100R	Br	Br, Brb100R
Lead Coated, (1.83 oz per sq ft)	2	...	10PHR	25R—Brb60R	10PHY	37R	33SR	4R	25SR

At each location more than 900 specimens were exposed. These included short lengths of wire (42 in. long) and wire strand at all locations; farm field fence at nine sites; barbed wire at eight locations, and chain link fence at eight locations.

The extent of corrosion is being measured by visual examinations of all specimens at the test sites and by tension tests on the wire specimens. The specimens at Bridgeport, Conn., and State College, Pa., were inspected in the spring of 1955 by a traveling committee. Inspections were made at the other exposure locations by the university inspectors in charge of the exposure plots.

The summarized inspection records of corrosion through 1955 are shown in Table A for wire specimens, wire strand and chain link fence, and in Table B for farm field fence and barbed wire.

No wire specimens were removed for tension tests during the year because inspection of previous results showed that losses in strength were not sufficient to warrant further removals at this time.

Respectfully submitted on behalf of the Wire Test Inspection Committee,

FRED M. REINHART,
Chairman.

REPORT OF COMMITTEE A-7

ON

MALLEABLE IRON CASTINGS*

Committee A-7 on Malleable Iron Castings held two meetings during the year: at Atlantic City, N. J., on June 29, 1955, and at Buffalo, N. Y., on February 29, 1956.

The committee lost by death the services of Russell J. Anderson, a valuable and highly respected member who represented the Belle City Malleable Iron Co.

The committee consists of 54 members, of whom 27 are classified as producers, 15 as consumers, and 12 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, W. A. Kennedy.

Vice-Chairman, W. M. Albrecht.

Secretary, J. H. Lansing.

At the June meeting, the committee took action to recommend the exposure of malleable specimens at various locations supervised by the Advisory Committee on Corrosion.

The committee is considering the establishment of test requirements for T bolts and nuts for mechanical pipe joints.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Chemical and Physical Methods of Testing (L. G. Toye, chairman) is considering the development of a single standard method for the determination of yield strength in response to a request from the ASME Boiler and Pressure Vessel Committee.

The committee noted with interest the issuance by the National Bureau of Standards of their white iron reference sample No. 3 as a tentative standard. This is a signal accomplishment as no white iron reference material has been available previously.

This report has been submitted to letter ballot of the committee, which consists of 54 members; 35 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. A. KENNEDY,
Chairman.

JAMES H. LANSING,
Secretary.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

REPORT OF COMMITTEE A-9

ON

FERRO-ALLOYS*

Committee A-9 on Ferro-Alloys held its last meeting on June 15, 1954, in Chicago, Ill., during the Annual Meeting of the Society. A meeting is planned during the 1956 Annual Meeting of the Society in Atlantic City, N. J.

The officers elected for the ensuing term of two years are as follows:

Chairman, S. W. Poole.
Vice-Chairman, E. A. Lucas.
Secretary, W. H. Mayo.

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends that the Tentative Specification for Ferromanganese (**A 99 – 53 T**)[1] be continued without revision.

Respectfully submitted on behalf of the committee,

W. C. BOWDEN,
Chairman.

J. J. CROWE,
Secretary.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] 1955 Book of ASTM Standards, Part 1.

REPORT OF COMMITTEE A-10

ON

IRON-CHROMIUM, IRON-CHROMIUM-NICKEL, AND RELATED ALLOYS*

Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel, and Related Alloys held two meetings during the year: in Atlantic City, N. J., on June 29, 1955, during the Annual Meeting of the Society, and on February 29, 1956, at Buffalo, N. Y., during ASTM Committee Week.

The committee consists of 96 members, of whom 39 are classified as producers, 36 as consumers, and 16 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, Jerome Strauss.
Vice-Chairman, L. L. Wyman.
Secretary, M. A. Cordovi.

A new Subcommittee XIII has been formed with M. A. Cordovi, chairman, to cover the interests of the committee in the field of nuclear power plant equipment. The scope of the subcommittee is (*a*) to review and recommend revisions which may be required in applicable standards currently under the jurisdiction of the committee in the light of special requirements for nuclear installations, and (*b*) to determine whether sufficient data are available on which to base recommendations for new standards which may be required in this field.

REVISION OF TENTATIVE

The committee, jointly with Committee A-1 on Steel, recommends that the Tentative Specification for Seamless and Welded Austenitic Stainless Steel Pipe (**A 312 – 55 T**)[1] be revised. The revisions are set forth in the Report of Committee A-1.

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee, jointly with Committee A-1 on Steel, recommends for immediate adoption revisions in the following standards, and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Specifications for:

Seamless and Welded Austenitic Stainless Steel Tubing for General Service (**A 269 – 55**),[1] and
Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (**A 271 – 55**).[1]

The revisions are set forth in the Report of Committee A-1.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee IV on Methods of Corrosion Testing (O. B. Ellis, chairman) is considering the adoption as standard of the Tentative Recommended Practice for Descaling and Cleaning Stainless Steel Surfaces (A 380 – 54 T) with changes in the concentration of the nitric acid.

Under the chairmanship of C. P. Larrabee, a task group has made an inspection of the special stainless steel specimens which have been exposed at atmospheric test locations at Pittsburgh and New York City. A report of this inspection is appended hereto.[2]

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] 1955 Book of ASTM Standards, Part 1

A task group under the chairmanship of F. K. Bloom, appointed to survey current research programs on stress corrosion of stainless steels, has prepared a questionnaire for circulation in the near future. Technical Committee T-5 E of the National Association of Corrosion Engineers will distribute a similar questionnaire within NACE. The groups in the two societies are cooperating fully and the replies from their respective memberships will be correlated.

Progress has been made in procuring specimen materials for the atmospheric corrosion test program on stainless steels expected to be under way by the latter part of 1956.

Subcommittee V on Mechanical Testing (R. Heyer, chairman).—At the June 29, 1955, meeting, the Task Group on Tension Testing reported on tension tests of light gage type 301 sheets in three tempers, using strain-pacing type extensometers to control the speed of testing. For the annealed grade a decrease in strain rate from 0.5 to 0.3 in. per in. per min gave an increase of 5 per cent in tensile strength. Mill tests using a speed of testing in accordance with present test methods gave a tensile strength 13 per cent higher than at the controlled strain rate of 0.5 in. per in. per min. This demonstrates further the need for better definition of limits for speed of testing in product specifications for the speed-sensitive grades. A further program was initiated to procure the data necessary to make recommendations.

The subcommittee was unable to obtain additional test data on elongation of 8-in. gage length plate tension tests of stainless steels, and therefore it has been recommended that Subcommittee IX select specification limits for elongation in 8-in. for Specifications A 167, A 176, and A 240 from data already submitted. As this flat-plate specimen comes into use, data for possible revision of the limits will become available.

Subcommittee VI on Metallography (R. Franks, chairman) will study further the occurrence and identification of sigma phase in the nickel-chromium-iron stainless steels. It is expected that a task group set up for the purpose, among other approaches to the problem of identification, will consider the use of the electron microscope. A study is to be undertaken to determine by metallographic examinations the structure which may be characteristic of types 347 and 321 stainless steels which have been prone to cracking in large welded pipe used in the main steam lines of power generating plants.

Subcommittee VIII on Specifications for Wrought Products (R. B. Gunia, chairman).—A task group has been appointed to develop specifications covering AISI Types 201 and 202 alloys.

Subcommittee IX on Specifications for Flat Products (G. W. Hinkle, chairman).—A task group, under the chairmanship of W. F. Hodges, has been appointed to revise Specification A 240 in order to meet Boiler Code requirements.

The mechanical properties of AISI Type 410 (low-carbon), 305, and 348 stainless steels and AISI shearing tolerances in Specifications A 240, A 167, and A 176 are being balloted in the subcommittee. The AISI chemical composition ranges will be incorporated in specifications A 167, A 176, and A 177. A task group is preparing material specifications for AISI Types 201 and 202 alloys as related to flat products.

Subcommittee XII on Specifications for High-Temperature Super Strength

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[2] See p. 143.

Alloys (L. L. Wyman, chairman) selected three types of alloys from the Compilation of Chemical Compositions and Rupture Strengths of Super-Strength Alloys (*ASTM STP 170*) to form the basis of specifications, and subgroups as indicated have been formed:

Subgroup A on Hot-Cold Worked Alloys (16-25-6).

Subgroup B on Precipitation Hardenable Alloys (A 286 alloy).

Subgroup C on Cast Alloys (N-155).

The proposed definition of "super alloy" has been referred back to the task group on scope to determine the status of such alloys as 17-7-PH, 17-4-PH, AM-350, and Stainless W. The task group will determine how the close-packed hexagonal structure of cobalt-base alloys fits into the definition, and whether separate consideration should be given to magnetic *versus* non-magnetic rather than ferritic *versus* austenitic alloys.

An attempt will be made to coordinate the activities of the Investment Casting Institute's Committee on Casting Specifications with the work of Subgroup C.

The task group on alloy compilation will compile a list of martensitic super alloys similar to the austenitic alloy compilation in *ASTM STP 170*.

Joint A-1, A-10 Subcommittee on Stainless Tubing Specifications (J. J. B. Rutherford, chairman) has approved during the year (*a*) specifications for welded large O. D. stainless pipe, and (*b*) the inclusion of types 347 and 348 in the appropriate specifications (these grades will be appropriately identified in the next revision of Specifications A 312), (*c*) increasing the phosphorus limit to 0.04 per cent max in all tubular stainless grades, (*d*) adding grades 304 ELC and 316 ELC to the appropriate tubular-product specifications and the inclusion of these grades with a 0.04 per cent maximum limit on carbon, (*e*) increasing the maximum silicon content in all stainless tubular product specifications, except Specifications A 376, from 0.75 per cent limit, to 1.00 per cent, and (*f*) inclusion of mandatory heat-treating clauses in the stainless tubular product specifications (A 249, A 269, and A 312).

Preliminary consideration has been given to the request from the ASME Boiler and Pressure Vessel Committee for inclusion of a proprietary Cr-Ni-Mo-Cu-Cb-Ta stainless alloy in Specification A 312.

Respectfully submitted on behalf of the committee,

JEROME STRAUSS,
Chairman.

M. A. CORDOVI,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee A-10, jointly with Committee A-1 on Steel, presented to the Society through the Aministrative Committee on Standards the following recommendations:

Withdrawal of Emergency Alternate Provisions for:

Seamless and Welded Austenitic Stainless Steel Tubing for General Service (EA – A 269),
Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (EA – A 271), and

Seamless and Welded Austenitic Stainless Steel Pipe (EA – A 312).

These recommendations were accepted by the Standards Committee on November 21, 1956.

APPENDIX I

REPORT OF TASK GROUP ON INSPECTION OF NICKEL-BEARING AND NONNICKEL-BEARING STAINLESS STEELS

During March, 1956, the task force made the third annual inspection of the sheets of nine grades of stainless steel, each in No. 2B and No. 4 finish. As in 1955, somewhat more dirt was observed on the sheets at Pittsburgh than on those at New York City.

The left half of each sheet was cleaned by gently rubbing with whiting spread onto a cloth wet with water. Most of the whiting was removed with water and the balance with dry cloths.

At Pittsburgh there has been some rusting of the groundward side of many specimens during the last year. This is particularly the case on the 2-in. wide flange that is used to attach the upper half of the sheet to the lower half by spot-welding, and by machine screws and nuts. All spot welds at Pittsburgh were stained more than those at New York.

It was noticeable that the minor differences in texture of the 2B finish caused more differences in general appearance than did the differences in composition of the various steels.

On the groundward surfaces at Pittsburgh the ascending order of excellence was Type 410, 16-1-17, 13-1-18, Type 430, with Types 442, 446, 301, and 304 being about the same, and with Type 201 having the least staining. As seen in the table, the groundward surfaces of many of the sheets at New York were "OK."

The detailed results of the inspections are given in Tables I and II,

C. P. Larrabee,
Task Force Chairman.

TABLE I.—INSPECTION OF STAINLESS STEEL PANELS AT PITTSBURGH, PA.
EXPOSED: MAY 20, 1953. INSPECTED: MARCH 26, 1956.

Material	Finish	Before and After Cleaning							Spot Welds	Bolt Holes
		General Appearance			Gutter		Faying Surfaces			
		Skyward		Groundward						
		Before	After	Before	Before	After	Before	After		
Type 410	No. 4	Tr Y	Tr Y, P	30% R	Y	Y, P	R	R	Y	Y
Type 410	No. 2B	Tr Y	Tr Y, P	30% R	Y	Y, P	R	R	Y	Y
Type 430	No. 4	No Y	OK	Tr R(1)	OK	OK	OK	OK	Tr Y	Tr Y
Type 430	No. 2B	No Y	OK	Tr R(1)	OK	OK	NI	NI	Tr Y	NI
Type 442	No. 4	No Y	OK	Tr R(1)	OK	OK	NI	NI	Tr Y	NI
Type 442	No. 2B	No Y	OK	Tr R(1)	OK	OK	NI	NI	Tr Y	NI
Type 446	No. 4	No Y	OK	Tr R(1)	OK	OK	NI	NI	Tr Y	NI
Type 446	No. 2B	No Y	OK	Tr R(1)	OK	OK	NI	NI	Tr Y	NI
Type 301	No. 4	No Y	OK	Tr R(1)	OK	OK	NI	NI	Tr Y	NI
Type 301	No. 2B	No Y	OK	Tr R(1)	OK	OK	NI	NI	Tr Y	NI
Type 304	No. 4	No Y	OK	Tr R(1)	OK	OK	NI	NI	Tr Y	NI
Type 304	No. 2B	No Y	OK	Tr R(1)	OK	OK	NI	NI	Tr Y	NI
Type 201	No. 4	No Y	OK	OK	OK	OK	NI	NI	Tr Y	NI
Type 201	No. 2B	No Y	OK	OK	OK	OK	NI	NI	Tr Y	NI
16-1-17	No. 4	No Y	OK	10% R	OK	OK	NI	NI	Tr Y	NI
16-1-17	No. 2B	No Y	OK	10% R	OK	OK	NI	NI	Tr Y	NI
13-1-18	No. 4	No Y	OK	5% R	OK	OK	OK	OK	Tr Y	OK
13-1-18	No. 2B	No Y	OK	5% R	OK	OK	OK	OK	Tr Y	OK

OK—No visible attack
Y—Yellow stains
R—Red rust
P—Tiny pits
Tr—Trace

NI—Not inspected
%—As prefix to Y or R indicates percentage of surface area so affected
(1)—Particularly the exposed flange

TABLE II.—INSPECTION OF STAINLESS STEEL PANELS AT NEW YORK, N. Y.
EXPOSED: JULY 10, 1953. INSPECTED: MARCH 12, 1956.

Material	Finish	Before and After Cleaning							Spot Welds	Bolt Holes	Remarks
		General Appearance			Gutter		Faying Surfaces				
		Skyward		Groundward							
		Before	After	Before	Before	After	Before	After			
Type 410	No. 4	P&Y—S	P&Y	25% R (upper ½) 50% R (lower ½)	P&Y	P	Y&R	OK	R	Y&R (3)	Many pits on skyward surface. Yellow stains and dirt present, but no red rust on either panel.
Type 410	No. 2B	P&Y—S	P&Y	R	P&Y	P	OK	OK	R	R (2)	
Type 430	No. 4	OK—S	OK	P, Y to R	Y	OK	Y	OK	R	OK	Stain beneath washer on top surface of No. 4 finish panel. Red-to-yellow stains on top surface along north & east edges reaches in ⅛ in. on both Type 430 panels.
Type 430	No. 2B	OK—S	OK	P, Y to R	Y	OK	NI	NI	*	NI	
Type 442	No. 4	OK—S	OK	P, Y to R	OK	OK	NI	NI	*	NI	Stains on top surface along north and east edges of both panels have progressed to as much as ¼ in. from edges.
Type 442	No. 2B	OK—S	OK	P, Y to R	OK	OK	NI	NI	*	NI	
Type 446	No. 4	OK	OK	OK**	OK	OK	NI	NI	*	NI	
Type 446	No. 2B	OK—S	OK	OK	OK	OK	NI	NI	*	NI	
Type 301	No. 4	OK	OK	OK	OK	OK	NI	NI	*	NI	
Type 301	No. 2B	OK	OK	OK	OK	OK	NI	NI	*	NI	
Type 304	No. 4	OK	OK	OK	OK	OK	NI	NI	*	NI	
Type 304	No. 2B	OK	OK	OK	OK	OK	NI	NI	*	NI	
Type 201	No. 4	OK	OK	OK	OK	OK	NI	NI	*	NI	
Type 201	No. 2B	OK	OK	OK	OK	OK	NI	NI	*	NI	
16-1-17	No. 4	OK—S	OK	R	Y	OK	NI	NI	R	NI	Stain along east edge of one of these. Skyward surface of these is not quite so good in appearance as Type 430.
16-1-17	No. 2B	OK	OK	R	Y	OK	NI	NI	R	NI	
13-1-18	No. 4	P&Y—S	OK	10% R (upper ½) 60% R (lower ½)	Y	OK	Y to R	OK	R	Y to R (5)	Some staining on east edge of top surface of both of these. These panels are between Types 410 and 430 in appearance.
13-1-18	No. 2B	P&Y	OK	10% R (upper ½) 50% R (lower ½)	Y	OK	Y	OK	R	Y(1)	

OK—No visible attack.
Y—Yellow stains.
R—Red rust.
P—Tiny pits.

S—Stain or stains, yellow to red, along north or east edges, or both.
NI—Not inspected.
%—As prefix to Y or R indicates percentage of surface area so affected.
( )—As suffix indicates number of bolt holes so affected.
*Spot weld about the same in appearance as groundward surface.
**Suspicious area on underside of Section 2 panel. Looks like small cracks, plus yellow-to-red stains. One rust spot at east edge of 2B panel.

REPORT OF COMMITTEE B-1

ON

WIRES FOR ELECTRICAL CONDUCTORS*

Committee B-1 on Wires for Electrical Conductors held three meetings during the year: in Washington, D. C., on October 25, 1955; in New York, N. Y., on January 13, 1956; and in Roanoke, Va., on March 20, 1956. Meetings of Subcommittees II, IV, and VII also were held during the year.

The officers elected for the ensuing term of two years are as follows:

Chairman, D. Halloran.
Vice-Chairman, W. R. Hibbard.
Secretary, A. A. Jones.

The members-at-large elected to the Advisory Subcommittee are as follows: L. B. Curtis, A. A. Defoe, M. F. W. Heberlein, W. F. Markley, and E. M. Wolf.

The subcommittee organization and their chairmen remain unchanged.

The committee now consists of 82 members, of whom 41 are classified as producers, 28 as consumers, and 13 as general interest members.

Revisions of Tentatives

The committee recommends that the following six specifications be revised as indicated and continued as tentative:

Tentative Specifications for Tinned Soft or Annealed Copper Wire for Electrical Purposes (B 33 – 53 T):[1] Revise as appended hereto.[2]

Tentative Specifications for Lead-Coated and Lead-Alloy-Coated Soft Copper Wire for Electrical Purposes (B 189 – 53 T):[1] Revise as appended hereto.[2]

Tentative Specifications for Concentric-Lay-Stranded Aluminum Conductors, Steel-Reinforced (ACSR) (B 232 – 55 T):[1]

Section 1 (*b*).—Change to read as follows:

> For the purpose of these specifications, conductors are classified as follows:
>
> *Class AA*. For bare conductors usually used in overhead lines. These conductors are divided into two types as follows:
>
> (*1*) Conductors used for regular overhead line construction, and
>
> (*2*) Conductors having a high ratio of mechanical strength to current-carrying capacity used for overhead ground wires and for extra-long span construction.
>
> *Class A*. For conductors to be covered with weather-resistant (weatherproof) materials.

Section 2.—Change Item (*1*) to read: "Quantity of each size, stranding, and class."

Add a new Item (*4*) to read: "(*4*) Type of steel core wire and weight (standard, B or C) of coating (Section 3 (*b*))," and renumber the subsequent Items accordingly.

Section 3 (*b*).—Add the following clause to the end of this section: "except that if so specified in the purchase order or contract, the requirements of Tentative Specifications for Zinc Coated (Galvanized) Steel Core Wire (With Coatings Heavier than Standard Weight) For Aluminum Conductors, Steel-Reinforced (ACSR) (ASTM Designation:

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] 1955 Book of ASTM Standards, Part 2.

[2] The revised tentative appears in the 1956 Supplement to Book of ASTM Standards, Part 2.

B 261) for wire of the specified coating weight shall apply."

Section 5.—At the beginning of Paragraphs (*a*), (*b*), (*c*), (*d*), and (*e*) insert the phrase "For class AA conductors, the preferred . . ."

TABLE I.—CONSTRUCTION REQUIREMENTS OF CONCENTRIC-LAY-STRANDED ALUMINUM CONDUCTORS, STEEL-REINFORCED, CLASS A.

(New Table II, B 232 - 55 T)

Conductor Size (Aluminum)		Stranding			
		Aluminum		Steel	
cir mils	Awg	Number of Wires	Diameter, in.	Number of Wires	Diameter, in.
211 600.....	0000	6	0.1878	1	0.1878
167 800.....	000	6	0.1672	1	0.1672
133 100.....	00	6	0.1490	1	0.1490
105 600.....	0	6	0.1327	1	0.1327
83 690.....	1	6	0.1182	1	0.1182
66 360.....	2	7	0.0974	1	0.1299
66 360.....	2	6	0.1052	1	0.1052
52 620.....	3	6	0.0937	1	0.0937
41 740.....	4	7	0.0772	1	0.1029
41 740.....	4	6	0.0834	1	0.0834
33 090.....	5	6	0.0743	1	0.0743
26 240.....	6	6	0.0661	1	0.0661

Add a new Paragraph (*f*) to read as follows, and reletter the subsequent paragraphs accordingly:

> (*f*) For class A conductors, the lay of the aluminum wires of aluminum conductors, steel-reinforced, having a single wire steel core and one layer of aluminum wires shall be not less than 8 nor more than 16 times the outside diameter of the conductor.

Section 6.—Change to read: "The number and diameter of aluminum and steel wires and the areas of cross-section of aluminum wires shall conform to the requirements prescribed in Tables I, II, and III (Notes 2 and 6)."

Table I.—Change the table heading to read "Construction Requirements of Concentric-Lay-Stranded Aluminum Conductors, Steel-Reinforced, Class AA."

New Table.—Add a new Table II to read as shown in the accompanying Table I; and renumber the subsequent tables accordingly.

Table II.—Change the heading for the present Table II to read "Construction Requirements of High-Strength Concentric-Lay-Stranded Aluminum Conductors, Steel-Reinforced, Class AA."

Section 10 (b).—To the end of this section add a reference to Note 8.

New Explanatory Note.—Add a new Explanatory Note 8 to read as follows:

> NOTE 8.—The increment of weight or electrical resistance of a completed concentric-lay-stranded conductor (k) is
>
> $$k = 100\,(m - 1)$$
>
> where m is the lay factor, and is the ratio of the weight or electrical resistance of a unit length of stranded conductor to that of a solid conductor of the same cross-sectional area or of a stranded conductor with infinite length of lay, that is, all wires parallel to the conductor axis. The lay factor m for the completed stranded conductor is the *numerical average* of the lay factors for each of the individual wires in the conductor, including the straight core wire, if any (for which the lay factor is unity). The lay factor (m_{ind}) for any given wire in a concentric-lay-stranded conductor is
>
> $$m_{\text{ind}} = \sqrt{1 + \frac{9.8696}{n^2}}$$
>
> $$\text{where } n = \frac{\text{Length of lay}}{\text{Diameter of helical path of the wire}}$$
>
> The derivation of the above is given in *Circular No. 31* of the National Bureau of Standards.

Tentative Specifications for Tinned Hard-Drawn and Medium-Hard-Drawn Copper Wire for Electrical Purposes (B 246 - 51 T):[1]

Section 2 (*a*).—Change "pure tin" to read "commercially pure tin."

Tentative Method for Determination of Cross-Sectional Area of Stranded Conductors (B 263 - 53 T):[1]

Section 7.—In Paragraphs (*a*), (*b*), and (*c*), change "*k*" to read "k = increment (increase) of weight and electrical resistance (from product specifications) in per cent." Also in Paragraph (*c*), change "ccs" to read "f_{ccs}."

TABLE II.—THICKNESS OF COATING.
(Revision of Table I, B 298 – 55 T)

Diameter, in.	Thickness of Coating, Microinches (For Information Only)			
	Class A, 1.25 per cent Silver	Class B, 2.50 per cent Silver	Class C, 4.00 per cent Silver	Class D, 6.10 per cent Silver
0.1285......	340	680	1 090	1 659
0.1144......	303	605	970	1 477
1.1019......	270	539	864	1 316
0.0907......	240	480	768	1 171
0.0808......	214	428	684	1 043
0.0720......	190	381	610	930
0.0641......	170	339	542	828
0.0571......	151	302	483	737
0.0508......	134	269	430	656
0.0453......	120	240	383	585
0.0403......	107	213	341	520
0.0359......	95	190	304	464
0.0320......	85	169	270	413
0.0285......	75	151	241	368
0.0253......	67	134	214	327
0.0226......	60	120	191	292
0.0201......	53	106	170	260
0.0179......	47	95	151	231
0.0159......	42	84	135	205
0.0142......	...	75	120	183
0.0126......	...	67	107	163
0.0113......	...	60	96	146
0.0100......	...	53	85	129
0.0089......	...	47	75	115
0.0080......	...	42	68	103
0.0071......	...	...	60	92
0.0063......	...	...	53	81
0.0056......	...	...	47	72
0.0050......	...	...	42	65
0.0045......	...	...	...	58
0.0040......	...	...	...	52
0.0035......	...	...	...	45
0.0031......	...	...	...	40

Tentative Specifications for Silver-Coated Soft or Annealed Copper Wire (B 298 – 55 T):[1]

Section 1.—Replace the second and third paragraphs with the following:

Class A.—Wire whose silver coating is at least 1.25 per cent of the total weight of the coated wire.

Class B.—Wire whose silver coating is at least 2.50 per cent of the total weight of the coated wire.

Class C.—Wire whose silver coating is at least 4.00 per cent of the total weight of the coated wire.

Class D.—Wire whose silver coating is at least 6.10 per cent of the total weight of the coated wire.

Table I.—Replace the columns entitled "Thickness of Coating" as shown in the accompanying Table II leaving other columns unchanged.

Section 4 (e).—Change to read as follows:

(*e*) *Weight of Coating.*—The weight of coating expressed in per cent of the total weight of the wire shall be not less than 1.25 per cent for class A; 2.50 per cent for class B; 4.00 per cent for class C; and 6.10 per cent for class D. When coatings other than these classes are required, the weight of the coating shall be not less than that specified. For ease of comparison, the thickness of coating for these classes has been included in Table I (Note 3).

Section 6 (d) (1).—Change the first sentence of the paragraph entitled "Treatment of Specimens" to read: "The specimens shall be thoroughly cleaned by immersion in a suitable organic solvent such as benzine, ether, or trichloroethylene for at least 3 min; then removed and wiped dry with a clean, soft cloth (**Caution:** See Note 8)."

Section 6 (d) (2).—Change the heading for this Item to read "*(2)* Special Solutions," retaining the present heading and text as the first paragraph.

Add the following new second paragraph under Item 2:

Hydrochloric Acid Solution (sp gr 1.088).—Commercial HCl (sp gr 1.12) shall be diluted with distilled water to a specific gravity of 1.088 measured at 15.5 C (60 F). A portion of the HCl solution having a volume of 180 ml shall be considered exhausted if it fails to remove within 15 sec the discoloration of the silver due to the polysulfide immersion.

Section 6 (d) (3).—Change the title "Immersion of Specimens" to read "Immersion in Polysulfide Solution."

Change the title of the second paragraph "Washing Specimens" to read "Washing." Add the following as the third paragraph:

Immersion in Hydrochloric Acid.—After washing, the specimen shall immediately be immersed for 15 sec in the HCl solution described in Paragraph (*d*) (*2*), thoroughly washed in clean water, and wiped dry wity a clean, soft cloth.

Explanatory Notes.—Add a new Note 8 to read as follows, renumbering the subsequent notes accordingly:

NOTE 8; **Caution.**—Consideration should be given to toxicity and flammability when selecting solvent cleaners.

The Note references in the body of the specifications also should be renumbered.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the Specifications for Soft or Annealed Coated Copper Conductors for Use in Hookup Wire for Electronic Equipment (**B 286 – 54 T**) be continued as tentative without revision.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following two specifications be approved for reference to letter ballot of the Society for adoption as standard without revision. These specifications have stood for two or three years as tentative, during which time no substantial criticism or suggestions for improvement have been received.

Tentative Specifications for:

Hard-Drawn Copper Wire (**B 1 – 53 T**),[1] and
Soft or Annealed Copper Wire (**B 3 – 54 T**).[1]

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions in the following six standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that these revisions may be referred to letter ballot of the Society:

Standard Specifications for Concentric-Lay-Stranded Copper Conductors, Hard, Medium-Hard, or Soft (B 8 – 53):[1]

Section 1 (*a*).—Add to the end of this Paragraph: "These conductors shall be constructed with a central core surrounded by one or more layers of helically laid wires."

Section 5 (*f*).—Change to read as follows:

(*f*) The direction of lay for conductors having a nominal cross-sectional area larger than No. 8 Awg shall be reversed in successive layers, unless otherwise specified by the purchaser.

Section 10 (*b*).—Add to the end of this section a reference to Note 9.

Section 11 (*a*).—Change "(Note 9)" to read "(Note 10)."

New Explanatory Note.—Add a new Explanatory Note 9 to read as recommended for the new Note 8 of Specifications B 232, renumbering subsequent notes accordingly.

Standard Specifications for Cored, Annular, Concentric-Lay-Stranded Copper Conductors (B 226 – 52):[1]

Section 10 (*b*).—Change "(Note 3)" to read "(Notes 3 and 7)."

New Explanatory Note.—Add a new Explanatory Note 7 to read as recommended for the new Note 8 of Specifications B 232.

Standard Specifications for Concentric-Lay-Stranded Copper Covered Steel Conductors (B 228 – 52):[1]

Section 9 (*b*).—To the end of this paragraph add a reference to Note 4.

Section 10.—At the end of this section change "(Note 4)" to read "(Note 5)."

New Explanatory Note.—Add a new Explanatory Note 4 to read as recommended for the new Note 8 of Specifications B 232, renumbering subsequent notes accordingly.

Standard Specifications for Concentric-Lay-Stranded Copper and Copper Covered Steel Composite Conductors (B 229 – 52):[1]

Section 9 (b).—To the end of this paragraph add a reference to Explanatory Note 6.

Table I.—Change the footnote reference (*a*) "See Note 6" to read "See Note 7."

Section 12 (a).—Change "(Note 7)" to read "(Note 9)."

New Explanatory Note.—Add a new Explanatory Note 6 to read as recommended for the new Note 8 of Specifications B 232 renumbering the subsequent notes accordingly.

Standard Specifications for Concentric-Lay-Stranded Aluminum Conductors, Hard-Drawn and Three-Quarter Hard-Drawn (B 231 – 55):[1]

Section 1 (a).—Add the following to the end of this Section: "These conductors shall be constructed with a central core surrounded by one or more layers of helically laid wires."

Section 5 (e).—Change to read as follows:

> (*e*) The direction of lay for conductors having a nominal cross-sectional area larger than No. 8 Awg shall be reversed in successive layers, unless otherwise specified by the purchaser.

Section 9 (b).—To the end of this paragraph add a reference to Note 7.

New Explanatory Note.—Add a new Explanatory Note 7 to read as recommended for the new Note 8 of Specifications B 232.

Standard Specifications for Three-Quarter Hard Aluminum Wire for Electrical Purposes (B 262 – 55):[1]

Section 10 (b).—Change the first sentence to read: "Joints may be made during final drawing or in the finished wire by electrical butt welding or by cold pressure-welding, with the following provisions."

Through an oversight, this action was inadvertently omitted from last year's report.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Editorial and Records (A. A. Jones, chairman) continued its editorial review of specifications and coordinated the various subcommittee recommendations regarding revisions of the various specifications.

Subcommittee II on Methods of Test and Sampling Procedures (R. H. Lloyd, chairman) completed revision of Specifications B 33 and B 189 incorporating statistical sampling procedures, and of Specifications B 8, B 226, B 228, B 229, B 231, and B 232 incorporating a method of calculating lay factor for concentric-lay-stranded conductors. It is continuing its efforts to develop a method of calculating lay factor for bunch-stranded conductors. After long deliberation, it concluded that an alternate method (to the bell method) of measuring elongation in 60-in. gage length (for HD and MHD wire) is not needed.

Subcommittee IV on Conductors of Copper and Copper Alloys (B. J. Sirois, chairman) assisted Subcommittee II in the revision of Specifications B 33 and B 189, completed revision of Specification B 8 incorporating provision for unilay stranding of small conductors, and completed revision of Specifications B 298 incorporating provision for 4 classes of silver-coated wire.

Task groups are analyzing data on commercial production of soft square and rectangular copper wire to be used as a basis for establishing acceptance limits when tested in accordance with the Tentative Method of Test for Stiffness of Bare Soft Square and Rectangular

Copper Wire for Magnet Wire Fabrication (ASTM Designation: B 279); cooperating with the U. S. Army Signal Corps Engineering Laboratories in the study of grey-tin transformation; cooperating with the Radio-Electronics-Television Manufacturers Association (RETMA) in an endeavor to coordinate specification requirements for electronic hookup wire; considering revision of corner radii requirements in Specification B 48 to permit improved quality of film-coated magnet wire; cooperating with Underwriters' Laboratories in an endeavor to coordinate length of lay requirements in small copper conductors; and considering the possibility of developing an industry standard test report form for reporting results of tests on bare conductors.

The subcommittee also cooperated with the National Bureau of Standards in the revision of NBS *Circular No. 31,* the fourth edition of which now is available from the Superintendent of Documents.

Subcommittee V on Conductors of Ferrous Metals (L. H. Winkler, chairman) has a task group considering preparation of proposed specifications for aluminum-coated steel core wire for ACSR Constructions.

Subcommittee VII on Conductors of Light Metals (P. V. Faragher, chairman) completed revision of Specifications B 231 incorporating provision for unilay stranding of small conductors, and of Specifications B 232 providing for two classes of conductors and three types of core wire.

Task groups are considering the preparation of proposed specifications for aluminum strap for magnet wire; preparation of proposed specifications for aluminum wire for communication (telephone) cable; revision of Specifications B 231 to permit stranding conductors from wire of a temper different from that of the completed conductors; and revision of Specifications B 232 to coordinate lengths of lay of the various steel core strandings.

This report has been submitted to letter ballot of the committee, which consists of 81 members; 56 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

D. Halloran,
Chairman.

A. A. Jones,
Secretary.

REPORT OF COMMITTEE B-2

ON

NON-FERROUS METALS AND ALLOYS*

Committee B-2 on Non-Ferrous Metals and Alloys met on June 28, 1955, in Atlantic City, N. J. Meetings of the Advisory Committee were held on June 28, 1955, in Atlantic City and on February 23, 1956, in New York City.

W. A. Mudge was appointed Secretary of the Committee to succeed G. H. LeFevre who had asked to be relieved of these duties.

E. H. Bunce and E. E. Schumacher were appointed as a special committee to handle research items for Committee B-2.

Sidney Rolle and W. E. Milligan have been appointed to the group responsible for participation by the United States in the work of Technical Committee 26 on Copper and Copper Alloys of the International Organization for Standardization.

The committee has had a very active group under the chairmanship of G. H. Harnden in arranging for a Symposium on Solder[1] at the 1956 Annual Meeting of the Society. The symposium will comprise four sessions.

A Symposium on Titanium[2] has been planned for the Second Pacific Area National Meeting to be held in September, 1956, in Los Angeles, Calif.

The officers elected for the ensuing two-year term are as follows:

Chairman, Bruce W. Gonser.

Secretary, W. A. Mudge.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following 12 specifications be continued as tentative without change:

Tentative Specifications for:

Nickel-Copper Alloy Plate, Sheet, and Strip (**B 127 - 49 T**),
Nickel Rods and Bars (**B 160 - 49 T**),
Nickel Seamless Pipe and Tubing (**B 161 - 49 T**),
Nickel Plate, Sheet and Strip (**B 162 - 49 T**),
Seamless Nickel and High Nickel Alloy Condenser, Evaporator and Heat-Exchanger Tubes (**B 163 - 49 T**),
Nickel-Copper Alloy Rods and Bars (**B 164 - 49 T**),
Nickel-Copper Alloy Seamless Pipe and Tubing (**B 165 - 49 T**),
Nickel-Chromium-Iron Alloy Rods and Bars (**B 166 - 49 T**),
Nickel-Chromium-Iron Alloy Seamless Pipe and Tubing (**B 167 - 49 T**),
Nickel-Chromium-Iron Alloy Plate, Sheet, and Strip (**B 168 - 49 T**),
Titanium Strip, Sheet, Plate, Bar, Tube, Rod, and Wire (**B 265 - 52 T**), and
Rosin Flux Cored Solder (**B 284 - 53 T**).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Refined Lead, Tin, Antimony, and Bismuth (Sidney Rolle, chairman).—A task force is considering the preparation of specifications and terminology for secondary lead. At present the forms of primary leads are covered in Specification B 29. The task force which is studying the classification of tin will report at the 1956 Annual Meeting.

Subcommittee VII on Refined Nickel and Cobalt, High-Nickel Alloys, and

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] Issued as separate publication *ASTM STP No. 189.*

[2] Issued as separate publication *ASTM STP No. 204.*

High-Cobalt Alloys Cast and Wrought (O. B. J. Fraser, chairman; E. R. Patton, secretary).—Specifications for two Hastelloy alloys in wrought and cast forms have been submitted to the subcommittee membership for letter ballot. For determining the yield strength of cast Hastelloy, the 0.2 per cent offset procedure will be used in place of the 0.5 per cent elongation under load.

Revisions in Specifications B 127, B 160, B 161, B 162, B 163, B 164, B 165, B 166, B 167, and B 168 have also been submitted to letter ballot of the subcommittee.

Subcommittee VIII on Miscellaneous Refined Metals and Alloys (E. E. Schumacher, chairman).—A coding system for titanium alloys is under investigation and will be submitted for consideration at the 1956 Annual Meeting. This subcommittee has also done considerable work in preparing for the titanium symposium at the West Coast Meeting in September, 1956.

This report has been submitted to letter ballot of the committee, which consists of 158 members; 78 members returned their ballots, of whom 76 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

BRUCE W. GONSER,
Chairman.

W. A. MUDGE,
Secretary.

REPORT OF COMMITTEE B-3
ON
CORROSION OF NON-FERROUS METALS AND ALLOYS*

Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys held two meetings during the year: in Atlantic City, N. J., on June 30, 1955, and in Buffalo, N. Y., on February 28, 1956.

The committee consists of 97 members, of whom 85 are voting members; 35 are classified as producers, 30 as consumers, and 20 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, K. G. Compton.
Vice Chairman, E. K. Camp.
Secretary, A. W. Tracy.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following test methods be continued as tentative without change:

Tentative Method of:

Total Immersion Corrosion Test of Non-Ferrous Metals (**B 185 - 43 T**),
Alternate Immersion Corrosion Test of Non-Ferrous Metals (**B 192 - 44 T**),
Salt Spray (Fog) Testing (**B 117 - 54 T**), and
Acetic Acid-Salt Spray (Fog) Testing (**B 287 - 54 T**).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee III on Spray Test (C. O. Durbin, chairman) plans to study further the factors affecting spray test results—the effect of metallic impurities in the salt; the range limits on fog collection; and the pH limits for the acetic acid-salt spray test.

Subcommittee VII on Weather (C. P. Larrabee, chairman).—The task group which is calibrating the corrosivity of the several ASTM test sites by means of zinc and steel specimens is continuing its work. The group studying the measurement of impurities in the atmosphere is trying to find some correlation between atmospheric pollution and results of the calibration tests.

Subcommittee VIII on Galvanic and Electrolytic Corrosion (G. V. Kingsley, chairman).—The assemblies of stainless steel disks coupled to other metals which have been on exposure 15 years will be brought in this year. The contact resistance of couples will be measured; then they will be visually examined, disassembled, cleaned, and weighed.

This report has been submitted to letter ballot of the committee, which consists of 85 voting members; 65 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

K. G. COMPTON,
Chairman.

A. W. TRACY,
Secretary.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17-22, 1956.

REPORT OF COMMITTEE B-4

ON

METALLIC MATERIALS FOR ELECTRICAL HEATING, ELECTRICAL RESISTANCE AND ELECTRICAL CONTACTS*

Committee B-4 on Metallic Materials for Electrical Heating, Electrical Resistance and Electrical Contacts held three meetings during the year: in Atlantic City, N. J., on June 27 and 28, 1955; in Philadelphia, Pa., on November 10 and 11, 1955; and in Pittsburgh, Pa., on March 8 and 9, 1956.

The present membership of Committee B-4 totals 53 members, of whom 22 are classified as producers, 24 as consumers, and 7 as general interest members.

At the June, 1955, meeting Subcommittee VIII was reorganized and set up as the new ASTM Committee F-1 on Materials for Electron Tubes and Semiconductor Devices. The title and scope of Committee B-4 were accordingly changed to the following:

Title.—Committee B-4 on Metallic Materials for Electrical Heating, Electrical Resistance and Electrical Contacts.

Scope.—Jurisdiction over the formulation of specifications and methods for testing metallic materials used in the construction of electrical heating devices, electrical resistance devices, electrical contacts, thermosensitive elements in thermostats, as well as the stimulation of research and standardization of nomenclature to accomplish the foregoing purposes.

The committee is sponsoring a Symposium on Electrical Contacts to be held at The Pennsylvania State University, University Park, Pa., during the week of June 24, 1956. A similar symposium was sponsored by the committee at this locale in 1954.

The officers elected for the ensuing term of two years are as follows:

Chairman, E. I. Shobert, II.
Vice-Chairman, Edward E. Edmunds.
Secretary, C. K. Strobel.
Assistant Secretary, C. L. Guettel.

The following were elected Honorary Members of the committee: P. H. Brace, F. E. Carter, and R. D. Van Nordstrand.

NEW STANDARDS, IMMEDIATE ADOPTION

The committee recommends that the Method of Test for Maximum Loading Stress at Temperature of Thermostat Metals (Cantilever Beam Method) be approved for reference to letter ballot of the Society for adoption as standard as appended hereto[1] and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society.

ADOPTION OF TENTATIVES AS STANDARD WITH REVISIONS

The committee recommends that the following tentatives be revised as appended hereto[1] and approved for reference to letter ballot of the Society for adoption as standard:

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] These tentatives appear in the 1956 Supplement to Book of ASTM Standards, Part 2.

Tentative Method of Test for:

Flexivity of Thermostat Metals (**B 106 - 51 T**),[2] and

Modulus of Elasticity of Thermostat Metals (Cantilever Beam Method) (**B 223 - 51 T**).[2]

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption the following revisions of the Standard Method of Test for Change of Resistance with Temperature of Metallic Materials for Electrical Heating (**B 70 - 39**)[2], and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Section 6 (*a*).—Change the second sentence to read as follows:

> The temperature of the furnace shall then be brought to the maximum specified temperature of test for the alloy in question and held at this value until the resistance of the specimen remains constant except for the relatively slow changes due to oxidation.

In line 12 change "200 C" to read "200 F."

WITHDRAWAL OF STANDARD

The committee recommends the withdrawal of the Standard Method of Test for Equivalent Yield Stress of Thermostat Metals (**B 191 - 50**).[2] This standard is being replaced by the new Method of Test for Maximum Loading Stress at Temperature of Thermostat Metals.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Electrical Heating and Resistance Materials (C. W. Armstrong, chairman).—Detailed changes are under consideration for Specifications B 267 and B 82 and also for the tentative revision of Method B 76. Studies are in progress on the temperature-resistance curves for 60 nickel, 15 chromium and 35 nickel, 20 chromium alloys; studies are also under way on an accelerated life test for iron-chromium-aluminum alloys.

Subcommittee II on Wrought and Cast Alloys for High Temperature Use (E. Edmunds, chairman) has been reviewing the standards under its jurisdiction and, in addition, has continued its investigations of deterioration of metals under high temperature conditions. An attempt is being made to define the various types of attack and after the definitions are secured, to work on each basic type of attack separately. Methods of measuring the rate of attack, which are realistic from the point of application, are also being considered.

Subcommittee III on Thermostat Metals (P. H. Brace, chairman) has prepared a new standard as reported above. Other work on application testing of various bimetal materials is being considered.

The subcommittee has recognized that the test methods for Resistivity of Metallically Conducting Resistance and Contact Materials (B 63), Change of Resistance with Temperature of Metallic Materials for Electrical Heating (B 70), and Diamond Pyramid Hardness of Metallic Materials (E 92), are applicable to the testing of thermostat metals.

Subcommittee IV on Contact Materials (J. D. Kleis, chairman):

Section A on Life Tests (T. K. Knapp, chairman).—The results of a considerable amount of work by various laboratories to correlate data on surety of make in contact materials have been analyzed statistically. The correlation at present is not satisfactory and a new approach is being considered.

Section B on Physical Properties (F. R. Farnham, chairman).—Tests on the method of measuring heat conductivity will be carried out shortly in various laboratories.

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[2] 1955 Book of ASTM Standards, Part 2.

Section C on Standardization of Contact Forms and Sizes (D. K. Wells, chairman).—Tables of dimensional standards for projection welding contacts have been agreed upon and submitted to the American Standards Association for consideration.

Section D on High Current Contacts (M. R. Swinehart, chairman) has completed collaborative tests on contact resistance of closed contacts with satisfactory results. A design of a machine is now being considered to measure the resistance of contact materials to damage by arcing.

Section E on Bibliography (E. I. Shobert II, chairman) has nearly completed the 1955 Supplement to the Bibliography on Electrical Contacts.

Section F on Microcontacts (A. L. VanEmden, chairman).—Interlaboratory tests using a special testing device to measure the reliability of microcontacts have not been in close agreement. Methods are being considered for revising the test and improving this correlation.

Consideration is being given to the desirability of the subcommittee undertaking work on plug-in contacts and sliding contacts.

This report has been submitted to letter ballot of the committee, which consists of 53 members; 39 members returned their ballots, of whom 33 voted affirmatively and 0 negatively.

Respectfully submitted in behalf of the committee,

E. I. SHOBERT II,
Chairman.

C. K. STROBEL,
Secretary.

REPORT OF COMMITTEE B-5

ON

COPPER AND COPPER ALLOYS, CAST AND WROUGHT*

Committee B-5 on Copper and Copper Alloys, Cast and Wrought, held two meetings during the year: at Philadelphia, Pa., on October 6, 1955, and at New York, N. Y., on January 26, 1956. At these two sessions the Executive Subcommittee and Subcommittees W-1, W-2, W-3, W-4 and G-1 also met.

During the year, 12 members were added to the committee and there were 15 removals. At the present time the committee consists of 131 members, of whom 108 are voting members; 49 are classified as producers, 40 as consumers, and 19 as general interest members.

Officers elected for the ensuing two-year term are as follows:

Chairman, G. H. Harnden.

First Vice-Chairman, W. D. France.

Second Vice-Chairman, William Romanoff.

Secretary, L. H. Adam.

A paper entitled "The Mechanical Properties of Wrought Phosphor Bronze Alloys" by G. R. Gohn and J. P. Guerard of Bell Telephone Laboratories, Inc. and H. S. Freynik of the Riverside Metal Co. was presented at the October meeting of the committee, and has been published by ASTM as *STP No. 183.*

RECOMMENDATIONS AFFECTING STANDARDS

The committee is submitting one new tentative specification, revisions in two tentative specifications, revisions in four standard specifications for immediate adoption, and the adoption of two tentatives as standard. The standards and tentatives affected, together with the revisions recommended, are covered in detail in the Appendix to this report.[1]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee W-1 on Plate, Sheet, and Strip (A. E. Moredock, chairman) recommended revisions for immediate adoption in Standard Specification B 36, prepared by a task group (M. D. Helfrick, chairman).

Among the subjects being considered by the subcommittee are the preparation of a specification for tin-coated copper; the revisions of "Basis of Purchase" sections; the limitations in plate size for Specification B 100; the revision of chemical requirements for alloy No. 8 in Specification B 122; the application of special thickness tolerances for copper-beryllium alloy to other materials in Specification B 248; the revision of mechanical properties, straightness tolerances, and grain-size requirements, and the addition of a hardness conversion table in Specification B 194; and the revision of chemical and physical requirements in Specification B 103.

Subcommittee W-2 on Rods, Bars, and Shapes (J. D. MacQueen, chairman) recommended that Specification B 283 be adopted as standard without revision

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*** Presented at the Fifty-ninth Annual Meeting of the Society, June 18–22, 1956.**

[1] See p. 161.

[2] The letter ballot vote on these recommendations was favorable, the results of the vote are on record at ASTM Headquarters.

and that Specification B 133 be continued as tentative without revision.

The subcommittee is considering a number of subjects including the clarification of requirements for test specimens in Specification B 139, the preparation of requirements for leaded brass rods of other than the free-cutting brass in Specification B 16, and the revision of chemical requirements for rod of type OF copper.

Subcommittee W-3 on Wire and Wire Rod (Sidney Metzger, chairman) recommended revisions for immediate adoption in Standard Specification B 206 and that Specification B 272 be continued as tentative without revision.

A task group (P. B. Tursi, chairman) prepared the tentative revisions for Specification B 206.

Some current projects of the subcommittee are the revision of chemical requirements, and provisions for stress-relief annealing alloy A spring wire in Specification B 159; and preparation of requirements for flat copper products with finished edges in sizes not provided for in Specification B 272.

Subcommittee W-4 on Pipe and Tube (G. C. Mutch, chairman) recommended that revisions be incorporated in Tentative Specifications B 251 and B 302. Revisions for immediate adoption were recommended in Standard Specifications B 111, and B 188. The subcommittee recommended the proposed new Tentative Specification for Copper Drainage Tube (DWV)[3] prepared by a task group (B. J. Sirois, chairman).

A task group (L. H. Adam, chairman) prepared the revision for Specification B 111. Another task group (B. J. Sirois, chairman) prepared the revisions for sampling requirements in Specifications B 251 and B 302.

Among the subjects under consideration in the subcommittee are the revision of hardness requirements in Specifications B 75, B 88, B 135 and B 188; for Specification B 111, the revision of requirements for the expanding test and for dimensional tolerances; preparation of a new specification for copper-silicon alloy pipe and tube; the revision of tensile requirements in Specification B 42 when the material is used in applications covered by the ASME Boiler and Pressure Vessel Code; preparation of requirements for 5S and 10S size schedules for pipe; revision of chemical requirements for type OF copper in Specification B 75; and the study of statistical sampling for Specification B 75 and other tube specifications.

Subcommittee F-1 on Castings, and Ingots for Remelting (G. H. Clamer, chairman; M. L. Steinbuch, secretary) recommended that Specification B 292 be adopted as standard without revision.

The subcommittee is working on the revision of chemical requirements in Specification B 198, the review of chemical compositions for the proper number of significant figures, and is assembling data on properties of cast copper-base alloys.

Subcommittee G-1 on Methods of Test (G. R. Gohn, chairman; J. P. Guerard, secretary) reviewed the changes in the tension test specimen for tubular products (Fig. 13 of Methods E 8) as proposed by Subcommittee 4 of Committee E-1 on Methods of Testing and recommended the adoption of the modified test specimen subject to the retention of a note controlling parallelism and taper.

The subcommittee called attention to the fact that it had not yet had an opportunity to determine whether the revised unified Tentative Methods for Determining the Average Grain Size of Metals (E 112) meets the needs of the copper and brass industry and asked Committee B-5 to discuss this officially matter with Committee E-4. Work is continuing on the preparation of a recommended practice on dimensional measurements, and on a study of the relation between the thickness of the test strip and the Rockwell hardness. The subcommittee is also reviewing all of the specifications under the jurisdiction of Committee B-5 to determine whether or not the various methods of test are specified in sufficient detail.

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[3] The new tentative appears in the 1956 Supplement to Book of ASTM Standards, Part 2.

Subcommittee G-3 on Editorial and Publications (W. F. Roeser, chairman) in cooperation with Subcommittee F-1 and the "W" subcommittees is reviewing the specifications under the jurisdiction of these subcommittees for editorial consistency. The subcommittee has been assigned the responsibility of fostering technical papers to be presented at meetings of Committee B-5 or the Society under the sponsorship of Committee B-5.

This report has been submitted to letter ballot of the committee, which consists of 108 voting members; 87 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

G. H. HARNDEN,
Chairman.

V. P. WEAVER,
Secretary.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS FOR COPPER AND COPPER ALLOYS, CAST AND WROUGHT

This Appendix contains the recommendations affecting certain standards covering copper and copper alloys which are referred to earlier in this report. The standards appear in their present form in the 1955 Book of ASTM Standards, Part 2.

NEW TENTATIVE

The committee recommends that the Specification for Copper Drainage Tube (DWV) be accepted for publication as tentative as appended hereto.[1]

REVISIONS OF TENTATIVES

The committee recommends that the following two tentative specifications be revised as indicated and continued as tentative:

Tentative Specification for General Requirements for Wrought Seamless Copper and Copper-Alloy Pipe and Tube (B 251 – 55 T):

Section 4.—In Paragraph (*a*) change "Portion Size" to read as follows:

Portion Size.—Sample pieces shall be taken for test purposes from each lot according to the following schedule:

Number of Pieces in Lot	Number of Sample Pieces to be Taken
1 to 50	1
51 to 200	2
201 to 1500	3
Over 1500	0.2 per cent of total number of pieces in the lot

Change Paragraph (*c*) to read as follows:

(*c*) *Physical Tests.*—For the physical tests, unless otherwise provided in the product specification, a specimen shall be taken from each of the pieces selected in accordance with Paragraph (*a*). The required physical test shall be made on each of the specimens so selected. In the case of Rockwell tests, the number of hardness readings shall not be less than three on each of the specimens.

Section 7.—Change Paragraph (*d*) to read as follows:

(*d*) If the results of any test made to determine the physical properties fails to meet the specified limits, this test shall be repeated on each of two additional specimens taken from different pieces and the results of both of these tests shall comply with the specified requirements.

In Paragraph (*e*), the first sentence, change "four" to read "additional."

Tentative Specification for Threadless Copper Pipe (B 302 – 55 T):

Section 9.—Change Paragraph (*a*) to read as follows:

(*a*) Sample pieces shall be selected for test purposes from each lot of 5,000 lb or fraction thereof, of each size according to the following schedule:

Number of Pieces in Lot	Number of Sample Pieces to be Taken
1 to 50	1
51 to 200	2
201 to 1500	3

Change Paragraph (*c*) to read:

(*c*) *Physical Tests.*—The required physical test shall be made on specimens taken from each sample piece selected. In the case of the Rockwell tests, the number of hardness readings shall not be less than three on each specimen.

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[1] The new tentative appears in the 1956 Supplement to Book of ASTM Standards, Part 2.

Section 12.—Change Paragraph (*b*) to read as follows:

(*b*) If the result of any test fails to conform to the requirements of Section 6, this test shall be repeated on two additional specimens taken from different pieces, and the result of both of these tests shall comply with the specified requirements.

In paragraph (*c*), the first sentence, change "four" to read "additional."

Revisions of Standards, Immediate Adoption

The committee recommends for immediate adoption revisions in the four specifications as indicated below and, accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that these revisions may be referred to letter ballot of the Society.

Standard Specification for Brass Plate, Sheet, Strip, and Rolled Bar (B 36 – 55):

New Section.—Add a new Section 2 to read as follows, renumbering subsequent sections accordingly:

2. *Ordering Information.*—Orders for material under this specification should include the following information:

(*1*) Quantity,

(*2*) Name of material: brass,

(*3*) Form of material: plate, sheet, strip or rolled bar,

(*4*) Alloy No. (Section 1(*a*)),

(*5*) Temper (Sections 5 and 6),

(*6*) Dimensions: thickness and width, and length if applicable,

(*7*) How furnished: rolls, stock lengths with or without ends, specific lengths with or without ends (Section 10 (*d*)),

(*8*) Type of edge, if required: slit-, sheared-, sawed-, rolled-edges square corners, rolled edges rounded corners, rolled rounded edges, rolled full rounded edges,

(*9*) Type of width and straightness tolerances, if required: slit metal tolerances, square-sheared metal tolerances, sawed metal tolerances, straightened or edge-rolled metal tolerances (Sections 10(*c*) and (*e*)),

(*10*) Specification number and date, and

(*11*) Special tests or exceptions, if any.

Section 3.—Delete the present Section 3 on "Basis of Purchase" and renumber subsequent sections accordingly.

TABLE I.—CROSS-SECTIONAL AREAS.

Pipe Size, in.	Cross-Sectional Area of Wall, sq in.[a]		
	Regular Pipe	Extra Strong Pipe	Type TP
¼	0.118	0.161	0.097
⅜	0.165	0.219	0.125
½	0.246	0.323	0.158
¾	0.335	0.440	0.201
1	0.471	0.648	0.255
1¼	0.694	0.893	0.326
1½	0.825	1.08	0.375
2	1.09	1.50	0.472
2½	1.58	2.28	0.574
3	2.26	3.05	0.891
3½	2.95	3.71	1.17
4	3.34	4.46	1.48
5	4.17	6.11	2.25
6	5.01	8.50	3.21
8	8.15	12.8	5.42
10	11.9	16.1	8.44
12	14.6	...	12.2

[a] 1 sq in. = 1,000,000 sq mils.

Standard Specification for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 – 54):

Section 10.—Insert the following after the second sentence: "In all other cases the test specimen shall include the finished tube end."

Standard Specification for Seamless Copper Bus Pipe and Tube (B 188 – 55):

Tables II and III.—Delete column headed "Cross-Sectional Area of Bore, sq in." and replace with a column headed "Cross-Sectional Area of Wall, sq in." as indicated in the accompanying Table I.

Standard Specification for Copper-Nickel-Zinc Alloy (Nickel Silver) Wire (B 206 – 54):

Section 1.—Add alloy E for general use, with nominal composition of 65 per cent copper, 10 per cent nickel, 25 per cent zinc.

Table I.—Add alloy E with chemical requirements as follows:

Copper, per cent	63.5 to 66.5
Nickel, per cent	9.0 to 11.0
Lead, max, per cent	0.05
Iron, max, per cent	0.25
Manganese, max, per cent	0.50
Zinc, per cent	remainder

Table II.—Change column heading "Alloy D" to read "Alloys D and E."

Table III.—Add "E" to each line under the column heading "Alloy."

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following two tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Specifications for:

Copper and Copper-Alloy Die Forgings (Hot Pressed) (**B 283 – 53 T**), and

Nickel-Tin Bronze Castings (**B 292 – 54 T**).

REPORT OF COMMITTEE B-6

ON

DIE-CAST METALS AND ALLOYS*

Committee B-6 held two meetings during the year: in Atlantic City, N. J. on June 29, 1955, and in Buffalo, N. Y., on February 29, 1956.

At the present time the committee consists of 95 members, of whom 48 are classified as producers, 24 as consumers and 23 as general interest members.

The officers and 6 members-at-large on the Advisory Committee elected for the ensuing term of two years are as follows:

Chairman, W. Babington.
Vice-Chairman, A. E. Weiss.
Secretary, G. L. Werley.
Advisory Committee: Members-at-large, F. C. Bennett
D. L. Colwell
R. F. Cramer
A. E. Martin
J. W. Meier
A. J. Prickett

REVISION OF TENTATIVES

The committee recommends that the following tentative be revised as indicated and continued as tentative:

Tentative Specifications for Aluminum-Base Alloy Die Castings (B 85 – 54 T):[1]

New Section.—Add a new Section 10 to read as follows:

> 10. The Appendix contains a table which shows certain casting and other outstanding characteristics which are usually considered in selecting a die-casting alloy for a specific application. The characteristics are rated from (1) to (5), (1) being the best and (5) being the least desirable alloy. In applying these ratings, it should be noted that all the alloys have sufficiently good characteristics to be accepted by users and producers of die castings. Hence a rating of (5) indicates a commercial alloy, although in certain cases its application may be limited or its manufacture may be restricted to relatively simple castings.

Appendix.—Add the accompanying Table I as an Appendix to the specifications.

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends that the Tentative Specification for Zinc-Base Alloys in Ingot Form for Die Castings (**B 240 – 52 T**)[1] be continued as tentative without revision.

EDITORIAL CHANGE

In order to correct an error, the committee recommends the following editorial change in the Standard Specifications for Copper-Base (Brass) Alloy Die Castings (**B 176 – 52**):[1]

Section 4.—In the Note, change the yield strength of Alloy ZS144A from "30,000 psi" to "50,000 psi."

The recommendations in this report have been submitted to letter ballot of

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17-22, 1956.

[1] 1955 Book of ASTM Standards, Part 2.

TABLE II.—DIE C ASTING AND OTHER CHARACTERISTICS.

Alloy	Approximate Melting Range, deg Fahr	Die Casting Characteristics			Other Characteristics									
		Resistance to Hot Cracking[a]	Pressure Tightness	Die Filling Capacity[b]	Normally Heat Treated[c]	Resistance to Corrosion[d]	Machining[e]	Polishing[f]	Electroplating[g]	Anodizing Appearance[h]	Chemical Oxide Coating[i] (Protection)	Strength at Elevated Temperature[j]	Suitable for Welding	Suitable for Brazing
G8A	995–1150	5	5	4	—	1	1	1	5	1	1	4	No	No
S5C	1065–1170	2	3	3	—	3	5	4	2	4	3	5	No	No
S12B	1065–1080	1	2	1	—	3	4	5	3	5	3	3	No	No
S12A	1065–1080	1	2	1	—	3	4	5	3	5	3	3	No	No
SC54B	990–1135	4	3	5	—	5	2	2	4	2	5	3	No	No
SC54A	990–1135	4	3	5	—	5	2	2	4	2	5	3	No	No
SC84A	1000–1100	2	2	2	—	4	3	3	1	3	5	2	No	No
SC84B	1000–1100	2	2	2	—	4	3	3	1	3	5	2	No	No
SG100A	1035–1105	1	1	1	—	2	3	3	1	3	3	2	No	No
SG100B	1035–1105	1	1	1	—	2	3	3	1	3	3	1	No	No
SC114A	960–1080	2	2	1	—	4	3	3	2	4	4	2	No	No

[a] Ability of alloy to withstand stresses from contraction while cooling through hot-short or brittle temperature range.

[b] Ability of liquid alloy to flow readily in die and fill thin sections.

[c] Heat treatments generally confined to low-temperature to provide stress relief or increased ductility. Treatments to improve properties not generally applicable.

[d] Based on resistance of alloy in standard type salt spray test.

[e] Composite rating based on ease of cutting, chip characteristics, quality of finish and tool life.

[f] Composite rating based on ease and speed of polishing and quality of finish provided by typical polishing procedure.

[g] Ability of the die casting to take and hold an electroplate applied by present standard methods.

[h] Rated on lightness of color, brightness and uniformity of clear anodized coating applied in sulfuric acid electrolyte.

[i] Rated on combined resistance of coating and base alloy to corrosion.

[j] Rating based on tensile and yield strengths at temperatures up to 500 F, after prolonged heating at testing temperature.

the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Aluminum-Base Die-Casting Alloys (E. V. Blackmun, chairman) reports that new mechanical properties have been determined for Alloys SG100A and SG100B but that these results as well as those reported a year ago for SC84A and SC84B will be reviewed further before being recommended for inclusion in the Appendix of Specifications for Aluminum-Base Alloy Die Castings (B 85).

Mechanical properties for Alloys S12A and S12B are being determined and similar tests are planned for Alloys SC114A, G8A and S5C.

Consideration is being given to changing the chemical requirement limits for 0.5 per cent or less to two significant places in Specification B 85 for the purpose of achieving better coordination with the Ingot Specification (B 179).

The subcommittee prepared the revisions in Specification B 85 - 54 T covered earlier in this report.

Approval is being sought for the removal of alloys SC-54A and SC-54B from Specification B 85 on the basis of near obsolescence due to commercial preference for the more castable alloys SC-84A and SC-84B.

Subcommittee II on Zinc-Base Die-Casting Alloys (A. E. Weiss, chairman).—

The recommendations made by the task group in the recording of the evolution of the present zinc die-casting alloys and the description of their relative differences are being put to separate letter ballots in an attempt to accelerate acceptance of at least some of the recommendations.

Subcommittee V on Exposure and Corrosion Tests (D. H. Kleppinger, chairman).—A task group has been appointed to prepare a report on the mechanical properties of aluminum Alloys IVa, V, and Va after 5, 10 and 20 years' exposure at Altoona, Pa., and New York City for publication by Committee B-6. The same task group will also compile the exposure test data for aluminum Alloys G8A, SG-100A, and SC-84A for publication in the ASTM BULLETIN.

The three-year exposure bars of the SC-84A alloy with controlled zinc contents are scheduled to be removed in April, 1956, from the New York Port Authority roof, and from the 80- and 800-ft sites at Kure Beach, N. C. Plans for testing these specimens have been completed.

The New York Port Authority roof has become an undesirable exposure site due to its proximity to a helicopter port. A new New York area site, in the U. S. Steel Supply Division grounds in Newark, N. J., has been made available to the subcommittee. A task group is to determine whether the SC84A specimens should be discarded or moved, and if moved, whether they should be cleaned before re-exposure at the new site.

Subcommittee VII on Magnesium-Base Die-Casting Alloys (V. D. Sweeney, chairman).—Consideration is being given to changing the chemical requirement limits for 0.5 per cent or less to two significant places in Table I of the Specifications for Magnesium-Base Alloy Die Castings (B 94 - 52).

Subcommittee VIII on Brass Die-Casting Alloys (J. C. Fox, chairman) has encountered a further delay in the determination of the mechanical properties for Alloy ZS331A.

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[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee IX on Die Casting Processes (A. E. Martin, chairman).—has completed its assignment and it has asked to be discharged in June, 1956.

This report has been submitted to letter ballot of the committee, which consists of 95 members: 93 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. BABINGTON,
Chairman.

GEORGE L. WERLEY,
Secretary.

REPORT OF COMMITTEE B-7

ON

LIGHT METALS AND ALLOYS, CAST AND WROUGHT*

Committee B-7 on Light Metals and Alloys held two meetings during the year: at Atlantic City, N. J., in June 1955, and at Buffalo, N. Y., in February 1956.

The committee consists of 87 members, of whom 85 are voting members; 39 are classified as producers, 31 as consumers, and 17 as general interest members.

During the year, R. L. Templin resigned from the committee and as chairman of Subcommittee V because of ill health; F. S. Mapes and J. C. Jones passed away.

The following appointments, including replacements, were made: J. C. Millson was appointed Chairman of Subcommittee V on Testing Light Metals to replace R. L. Templin. Representatives appointed on subcommittees of Committee E-1 were as follows: 2 on Speed of Testing, A. A. Moore to replace R. R. Moore; 3 on Elastic Strength of Materials, J. C. Millson to replace R. L. Templin; 5 on Compression Testing, J. C. Millson to replace R. L. Templin; 6 on Indentation Hardness, F. M. Howell to replace F. S. Mapes; 7 on Impact Testing, J. C. Millson to replace F. S. Mapes; and 16 on Thickness Measurement, H. D. Monsch. The representative appointed to serve on Committee E-12 on Appearance was Fred Keller and on Subcommittee III on Aluminum and Aluminum-Alloy Filler Metal of the ASTM-AWS Joint Committee on Filler Metal was C. B. Gleason to replace D. A. Lawless, who resigned. (Other representatives on the joint committee are P. V. Faragher and R. B. Smith.)

The officers elected for the ensuing term of two years are as follows:

Chairman, I. V. Williams.
Vice-Chairman, D. L. Colwell.
Vice-Chairman, E. C. Gleason.
Secretary, R. B. Smith.

Recommendations Accepted by the Administrative Committee on Standards

Subsequent to the 1955 Annual Meeting, Committee B-7 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specifications for:

Aluminum and Aluminum-Alloy Sheet and Plate for Pressure Vessel Applications (B 178 - 54 T),
Aluminum and Aluminum-Alloy Sheet and Plate (B 209 - 54 T),
Aluminum-Alloy Drawn Seamless Tubes (B 210 - 54 T),
Aluminum and Aluminum-Alloy Bars, Rods, and Wire (B 211 - 54 T),
Aluminum and Aluminum-Alloy Extruded Bars, Rods, and Shapes (B 221 - 54 T),
Aluminum-Alloy Drawn Seamless Tubes for Condensers and Heat Exchangers (B 234 - 54 T),
Aluminum-Alloy Extruded Tubes (B 235 - 54 T),
Aluminum-Alloy Pipe (B 241 - 54 T),
Aluminum-Alloy Die Forgings (B 247 - 54 T),
Aluminum and Aluminum-Alloy Bars, Rods, and Shapes for Pressure Vessel Applications (B 273 - 54 T), and
Aluminum and Aluminum-Alloy Pipe and Tube for Pressure Vessel Applications (B 274 - 54 T).

These recommendations were accepted by the Standards Committee on October 17, 1955, and the revised tentative specifications appear in the 1955 Book of ASTM Standards, Part 2.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

RECOMMENDATIONS AFFECTING STANDARDS

The committee recommends two new specifications for publication as tentative, revisions in 20 tentative specifications, revisions for immediate adoption in two standard specifications, the adoption of two tentatives as standard without revision, and the withdrawal of one standard specification. The standards and tentatives affected, together with the revisions recommended, are covered in detail in the Appendix to this report.[1]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Aluminum and Aluminum Alloy Ingots (R. A. Quadt, chairman) recommended withdrawal of Standard Specification for Aluminum Ingots for Remelting (B 24 – 46), which is not used because ingot is purchased to special requirements for each application; and revisions in the Standard Specification for Aluminum-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 179 – 55), as set forth in the Appendix to this report, to add two new alloys, to replace alloy ZC81A-B with ZC81A and ZC81B, to revise iron and magnesium limits for several alloys to obtain greater compatibility with the chemical requirements in B 26 and B 108 specifications for castings, and to make minor editorial changes.

Subcommittee II on Aluminum Alloy Castings (D. L. Colwell, chairman) prepared the revisions of the Tentative Specification for Aluminum-Base Alloy Sand Castings (B 26 – 55 T) and Tentative Specification for Aluminum-Base Alloy Permanent Mold Castings (B 108 – 55 T), as set forth in the Appendix to this report.

Subcommittee III on Wrought Aluminum and Wrought Aluminum Alloys (P. V. Faragher, chairman) prepared the new Tentative Specification for Aluminum-Alloy Drawn Seamless Coiled Tubes for Special Purpose Applications, the new Tentative Specification for Aluminum-Alloy Standard Structural Shapes, Rolled or Extruded, and revisions of the following 11 tentative specifications, as set forth in the Appendix to this report:

Specifications B 211, B 221, B 235, B 236, B 241, and B 273 are revised by adding a new section on identification marking. The same change is recommended for Specifications B 178, B 209, B 210, and B 274 as well as revisions in the chemical requirements for MG11A (AA3004), and in the tensile requirements for GM40A (AA5086) in B 178 and B 209, and in the dimensional tolerances for Schedules 5 and 10 pipe in B 274, to agree with current industry standards. The revisions in Specification B 234 consist of adding a new section on identification marking, and changing the permissible variations in dimensions to make them applicable to all alloys covered by the specification.

Subcommittee IV on Magnesium and Magnesium Alloys, Cast and Wrought (A. A. Moore, chairman) revised Standard Specification for Magnesium Ingot and Stick for Remelting (B 92 – 55) to make minor editorial changes, and revised the following tentative specifications, as set forth in the Appendix to this report: B 80, B 90, B 91, B 93, B 107, B 199, and B 217. These were revised to change the chemical requirements to express values of $\frac{1}{2}$ per cent and less to the one-hundredths place and values greater than $\frac{1}{2}$ per cent to the tenths place; to revise the section on basis of purchase; to add protective finishes to all except B 93; to add special inspection requirements to B 80, B 91, and B 199; to add additional dimensional tolerances to B 107 and B 217; to delete obsolete alloys AM80A and M1B, and add five new alloys and the T5 temper for AZ91C, to B 80; and to make minor editorial changes.

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[1] See p. 171.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee V on Testing Light Metals (J. C. Millson, chairman) developed the following note on rounding off tension test results and recommends that it be added to the tables of tensile requirements in specifications under the jurisdiction of the committee:

> NOTE.—For purposes of determining conformance with these specifications, each value for tensile strength and yield strength shall be rounded off to the nearest 100 psi, and each value for elongation shall be rounded off to the nearest 0.5 per cent, both in accordance with the rounding off method of the Recommended Practices for Designating Significant Places in Specified Limiting Values (ASTM Designation: E 29).

The subcommittee is continuing to investigate statistical sampling methods as applied to testing for mechanical properties and, in cooperation with Committee E-11 on Quality Control of Materials, is attempting to establish lot sizes to be used in defining a statistically satisfactory sampling frequency and method for application to the tensile requirements in specifications under the jurisdiction of Committee B-7.

Subcommittee VI on Anodic Oxidation of Aluminum and Magnesium Alloys (Fred Keller, chairman) recommends that the Tentative Method of Measuring Thickness of Anodic Coatings on Aluminum by Means of the Filmeter (B 244 - 49 T) be adopted as standard without revision. The task group appointed several years ago to develop specifications for anodic coatings on aluminum for various applications is being reactivated.

Subcommittee VII on Codification of Light Metals and Alloys, Cast and Wrought (R. B. Smith, chairman) recommends that the Tentative Recommended Practice for Temper Designation of Light Metals and Alloys, Cast and Wrought (B 296 - 54 T) be adopted as standard without revision.

Subcommittee VIII on Atmospheric Exposure Tests (L. H. Adam, chairman) has installed test specimens of 27 aluminum and 11 magnesium alloys at five ASTM atmospheric exposure test sites: on top of the Port of Authority Building, New York City, for industrial atmospheric conditions; at State College, Pa., for rural; at Kure Beach, N. C., for Atlantic Coast marine; at Point Reyes, Calif., for Pacific Coast marine; and at Freeport, Tex., for Gulf Coast marine atmospheric conditions. Some of the specimens have been exposed for over four years and others for two years. Specimens exposed for one year have been examined and tested, and those exposed for three years are now being examined and tested.

This report has been submitted to a letter ballot of the committee, which consists of 87 members; 54½ members returned their ballots, of whom 51½ have voted affirmatively and 0 negatively.

Respectively submitted on behalf of the committee,

I. V. WILLIAMS,
Chairman.

R. B. SMITH,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee B-7 presented to the Society through the Administrative Committee on Standards the recommendation that the proposed Specifications for Round Aluminum-Alloy Welded Tubes be accepted for publication as tentative. This recommendation was accepted by the Standards Committee on September 5, 1956, and the new tentative specifications appear in the 1956 Supplement to Book of ASTM Standards, Part 2, bearing the designation B 313 - 56 T.

APPENDIX

PROPOSED RECOMMENDATIONS AFFECTING STANDARDS FOR LIGHT METALS AND ALLOYS, CAST AND WROUGHT

In this Appendix are given the recommendations affecting certain standards covering light metals and alloys referred to earlier in this report. The standards appear in their present form in the 1955 Book of ASTM Standards, Part 2.

NEW TENTATIVES

The committee recommends that the following two specifications be accepted for publication as tentative as appended hereto:[1]

Specification for:

Aluminum-Alloy Drawn Seamless Coiled Tubes for Special Purpose Applications, and

Aluminum-Alloy Standard Structural Shapes, Rolled or Extruded.

REVISION OF TENTATIVES

The committee recommends that the following tentative specifications be revised as indicated and continued as tentative:

Tentative Specification for Aluminum-Base Alloy Sand Castings (B 26 - 55 T):

Table I.—For alloy GS42A add a limit of 0.15 per cent maximum for other elements, total. For alloy SC82A revise the titanium limit from "0.35" to read "0.25" per cent maximum. For alloy ZC81A revise the silicon limit from "0.35" to read "0.25" per cent maximum, and the limit for other elements, each, from "0.15" to read "0.10" per cent maximum. Correct alloy GZ61B to read ZG61B. Delete Footnote *a* and the table of former alloy designations and reletter subsequent footnotes accordingly.

Tentative Specifications for Magnesium-Base Alloy Sand Castings (B 80 - 55 T).

Section 2.—Renumber Item (*4*) as (*7*) and add new Items (*4*), (*5*), and (*6*) as follows:

(*4*) Casting drawing giving all necessary dimensions and showing amount of finish left for machining,

(*5*) Surface treatment and finish (Section 12),

(*6*) Special inspection requirements (Section 13(*c*)).

Section 12.—Add a new sentence to the present Section 12 to read: "Unless otherwise specified, the castings shall be chrome pickled prior to shipment. See Explanatory Note 4."

New Note 4.—Add a new Explanatory note reading as follows:

NOTE 4.—The chrome pickle affords measurable protection against corrosion and tarnish during shipment and storage of the castings. After pickling, the castings will be grey to bronze or yellow in color depending on alloy and condition. The chrome pickle is not recommended for castings containing metal inserts. Such castings should be ordered shipped without surface treatment or protected with a slushing compound.

Section 13.—Add a new Paragraph (*c*) as follows:

(*c*) Special inspection requirements such as simulated service, pressure testing, X-ray, or fluorescent penetrant must be stated on the order. Unless specific reference is made to the degree of discontinuity by reference to Radio-

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[1] These tentatives appear in the 1956 Supplement to Book of ASTM Standard, Part 2.

TABLE I.—CHEMICAL REQUIREMENTS.
(Revision of Table I, B 80 - 55 T)

Alloy	Magnesium, per cent	Aluminum, per cent	Manganese, per cent	Zinc, per cent	Thorium, per cent	Rare Earths, per cent	Zirconium, per cent	Silicon, max, per cent	Copper, max, per cent	Nickel, max, per cent	Other Impurities, max, per cent
AM100A.....	remainder	9.3 to 10.7	0.10 min	0.30 max	...	...	...	0.30	0.10	0.01	0.30
AZ63A......	remainder	5.3 to 6.7	0.15 min	2.5 to 3.5	...	...	...	0.30	0.25	0.01	0.30
AZ81A......	remainder	7.0 to 8.1	0.13 min	0.40 to 1.0	...	...	...	0.30	0.10	0.01	0.30
AZ91C......	remainder	8.1 to 9.3	0.13 min	0.40 to 1.0	...	...	...	0.30	0.10	0.01	0.30
AZ92A......	remainder	8.3 to 9.7	0.10 min	1.6 to 2.4	...	...	...	0.30	0.25	0.01	0.30
EK30A......	remainder	...	...	0.30 max	...	2.5 to 4.0	0.20 min	...	0.10	0.01	0.30
EK41A......	remainder	...	...	0.30 max	...	3.0 to 5.0	0.40 to 1.0	...	0.10	0.01	0.30
EZ33A......	remainder	...	...	2.0 to 4.0	...	2.5 to 4.0	0.50 to 1.0	...	0.10	0.01	0.30
HK31A......	remainder	...	...	0.30 max	2.5 to 4.0	...	0.40 to 1.0	...	0.10	0.01	0.30
HZ32A......	remainder	...	...	1.7 to 2.5	2.5 to 4.0	0.10 max	0.15 min	...	0.10	0.01	0.30
ZE41A......	remainder	...	0.15 max	3.5 to 5.0	...	0.75 to 1.75	0.40 to 1.0	...	0.10	0.01	0.30
ZH62A......	remainder	...	...	5.2 to 6.2	1.4 to 2.2	...	0.50 min	...	0.10	0.01	0.30
ZK51A......	remainder	...	...	3.6 to 5.5	...	...	0.50 to 1.0	...	0.10	0.01	0.30
ZK61A......	remainder	...	...	5.5 to 6.5	...	...	0.6 to 1.0	...	0.10	0.01	0.30

graphs for Inspection of Aluminum and Magnesium Castings (ASTM Designation E 98 – 53 T), acceptance standards shall be as agreed upon by the purchaser and the manufacturer.

Table I.—Revise as indicated in the accompanying Table I. Notes 1 and 2 and Footnote *a* will remain unchanged.

Table II.—Delete alloys AM80A and M1B and add alloys as indicated in the accompanying Table II.

TABLE II.—TENSILE REQUIREMENTS.
(Additions to Table II, B 80 – 55 T)

Alloy	Condition	Tensile Strength, min, psi	Yield Strength (0.2 per cent offset) min, psi	Elongation in 2 in., min, per cent
AZ81A-T4	(solution heat treated)	34 000	...	7
AZ91C-T5	(artificially aged only)	20 000	11 000	2
HK31A-T6	(solution heat treated and artificially aged)	27 000	13 000	4
HZ32A-T5	(artificially aged only)	27 000	13 000	4
ZE41A-T5	(artificially aged only)	28 000	16 000	4
ZH62A-T5	(artificially aged only)	35 000	22 000	4

TABLE III.—DATA FOR USE WITH EXTENSION-UNDER-LOAD METHOD.
(Additions to Table III, B 80 – 55 T)

Alloy	Condition	Typical or Minimum	Yield Strength, (0.2 per cent offset) min, psi	Unit Deformation, inch per inch of gage length
AZ81A-T4	(solution heat treated)	typical	11 000	0.0037
HK31A-T6	(solution heat treated and artificially aged)	minimum	13 000	0.0040
HZ32A-T5	(artificially aged only)	minimum	13 000	0.0040
ZE41A-T5	(artificially aged only)	minimum	16 000	0.0045
ZH62A-T5	(artificially aged only)	minimum	22 000	0.0054

Explanatory Note 1.—Delete alloys AM80A and M1B and add the following:

Alloy AZ81A.—This alloy has a specific gravity of about 1.80. It is used primarily in the solution heat treated condition. This alloy will produce castings having maximum soundness with moderate mechanical properties.

Alloy HK31A.—This alloy has a specific gravity of about 1.79. It is used in the solution heat treated and aged condition. It is recommended for elevated temperature use up to 600 F.

Alloy HZ32A.—This alloy has a specific gravity of about 1.83. It is used in the artificially aged condition. It is recommended for elevated temperature use up to 600 F.

Alloy ZE41A.—This alloy has a specific gravity of about 1.84. It is used in the artificially aged condition. It has better castability, better soundness, and lower strength than ZK51A. It is not intended for high temperature service.

Alloy ZH62A.—This alloy has a specific gravity of about 1.88. It is used in the artificially aged condition. This alloy combines high strength with good ductility and has better castability than alloys with similar strength properties.

Table III.—Delete alloys AM80A and M1B and add alloys as indicated in the accompanying Table III.

Table IV.—Delete alloys AM80A and M1B. Values for Table IV on the new alloys have been requested from the American Foundrymen's Society but are not available at this time.

Tentative Specifications for Magnesium-Base Alloy Sheet (B 90 – 55 T):

Section 2.—Renumber Item (*5*) as (*6*) and add a new Item (*5*) as follows:

"(*5*) Surface treatment (Section 13)."

TABLE IV.—CHEMICAL REQUIREMENTS.
(Revision of Table I, B 90 – 55 T)

Alloy	Magnesium, per cent	Aluminum, per cent	Manganese, min, per cent	Zinc, per cent	Silicon, max, per cent	Copper, max, per cent	Nickel, max, per cent	Iron, max, per cent	Calcium, max, per cent	Other Impurities, max, per cent
AZ31A............	remainder	2.5 to 3.5	0.20	0.6 to 1.4	0.30	0.05	0.005	0.005	0.30	0.30
M1A.............	remainder	...	1.2	...	0.30	0.05	0.01	...	0.30	0.30

Section 13.—Add the following new paragraph: "Unless otherwise specified the sheet shall be oiled, chrome pickled, or given an acetic nitrate pickle and oiled, as elected by the purchaser, prior to shipment. See Explanatory Note 3."

Explanatory Note 3.—Add a new Explanatory Note as follows:

Note 3.—Either the chrome pickle or the oil finish affords a measurable protection against tarnish and corrosion during shipment and storage of the sheet. The oil finish is frequently preferred by purchasers intending to perform forming or drawing operations on the sheet.

Table I.—Delete Footnote *a* renumbering *b* as *a* and revise as indicated in the accompanying Table IV.

Tentative Specifications for Magnesium-Base Alloy Forgings (B 91 – 55 T):

Section 2.—Change Item (*4*) to (*7*) and add the following:

(*4*) Drawing showing dimensions of all die forgings and for all hand forgings not simple discs, rounds, squares, or rectangles. The amount of stock left for machine finish should be indicated,

(*5*) Surface treatment (Section 11),

(*6*) Special inspection requirements (Section 12(*c*)).

Section 9.—Revise to read as follows:

9. One tension test specimen shall be taken to represent each 1000 lb or fraction thereof of each part number in the shipment or inspection lot.

Section 11.—Add a new paragraph reading: "Unless otherwise specified, the forgings shall be chrome pickled prior to shipment. See Explanatory Note 4."

Section 12.—Add a new Paragraph (*c*) to read as follows:

(*c*) Special inspection requirements such as inspection by ultrasonic reflection or by fluorescent penetrant, must be shown on the order. Acceptance standards for special inspection requirements shall be as agreed upon by purchaser and manufacturer.

Explanatory Note 1.—Delete the last sentence.

TABLE V.—CHEMICAL REQUIREMENTS
(Revision of Table I, B 91 – 55 T)

Alloy[a]	Zinc, per cent	Nickel, max, per cent	Silicon, max, per cent	Other Impurities, max, per cent
TA54A....	0.30 max	0.01	0.30	0.30
AZ31B....	0.6 to 1.4	0.005	0.30	0.30
AZ61A....	0.40 to 1.5	0.005	0.30	0.30
AZ80A....	0.20 to 0.8	0.005	0.30	0.30
ZK60A....	4.8 to 6.2	...	...	0.30

New Explanatory Note.—Add a new Explanatory Note 4 as follows:

Note 4.—The chrome pickle treatment increases the resistance to surface tarnish and corrosion during shipment and storage. After treatment, the forgings will be dull bronze to bright yellow in color. The color varies with alloy and condition.

Table I.—Delete Footnote *a*, changing the present *b* to *a* and revise values for zinc, silicon, nickel and other impurities as indicated in the accompanying Table V, leaving other columns unchanged.

Table II.—Delete properties and footnote reference *c* on ZK60A-T5 hand forgings and change footnote *b* to apply to all alloys in Table II. Revise footnote *b* to read as follows: "Applicable only to die forgings not more than 3 in. in thickness. The tensile requirements for hand forgings will be lower and as agreed upon by the purchaser and manufacturer."

Table III.—Delete the properties and all reference to ZK60A hand forgings.

Tentative Specifications for Magnesium-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 93 – 55 T):

Section 2.—Revise to read as follows:

2. Orders for ingot under these specifications shall include the following information:

(*1*) Quantity in pounds,

(*2*) Alloy (Section 3),

(*3*) Form, magnesium ingot is normally furnished in the following commercial forms unless otherwise specified in the contract:

Form	Approximate Size and Shape, in.	Approximate Weight, lb.
Ingot (3 notches)..	4 by 4½ by 28...	20.0

Table I.—Change the manganese content of M1B alloy to read: "1.3" instead of "1.30." Add a zero as a second digit to the right of the decimal point on the silicon contents of all the alloys in the table with the exception of AM100B.

Add a zero as a second digit to the right of the decimal point, on the value of "Other Impurities" for all of the alloys listed in Table I. Change the zinc range on AZ91A, AZ91B, and AZ91C from "0.5 to 0.9" to "0.45 to 0.9 per cent."

Delete footnote *a*, and renumber footnote *b* as *a*.

Tentative Specifications for Magnesium Alloy Bars, Rods, and Shapes (B 107 – 55 T):

Section 2.—Renumber Item (*5*) as (*7*), delete Item (*4*), and add Items (*4*), (*5*), and (*6*) as follows:

(*4*) Diameter (for rods), depth and width (for square cornered bars), distance across flats (for sharp cornered hexagonal or octagonal bars). Round cornered bars and shapes other than bar or rod require a drawing. (Section 12),

(*5*) Length (Section 12),

(*6*) Surface treatment (Section 13).

Section 12.—Delete Paragraph (*d*) and add the following:

(*d*) Variations in length shall not exceed the amount permitted by Table VII.

(*e*) Roughness of surface shall be not greater than permitted by Table VIII.

(*f*) Deviation from specified radius shall not exceed that permitted by Table IX.

(*g*) Curved surfaces, flat surfaces, and squareness of cut ends shall conform to the following:

(*1*) *Curved Surfaces*.—Allowable deviation from specified contour, 0.005 in. per in. of chord length, 0.005 in. min; not applicable to more than 90 deg of any arc.

(*2*) *Flat Surfaces*.—Allowable deviation from flat, 0.004 in. per in. of width, 0.004 in., min. Not applicable to surfaces of walls 0.250 in. or less in thickness which completely enclose a void.

(*3*) *Cut Ends*.—Allowable deviation from square: 1 deg.

Section 13.—Add a new paragraph as follows:

Unless otherwise specified, the extruded rod, bar, or shapes shall be oiled or chrome pickled, as elected by the purchaser, prior to shipment.

New Explanatory Note.—Add a new Explanatory Note 3 to read as follows:

NOTE 3.—Either the chrome pickle or the oil finish affords a measurable protection against tarnish and corrosion during shipment and storage of magnesium extrusions.

Table I.—Revise columns referring to zinc, silicon, calcium and other impurities as indicated in the following Table VI leaving notes and other columns unchanged.

Table III.—Delete "of Alloys AZ31B, AZ61A, AZ80A, and M1A" from heading.

TABLE VI.—CHEMICAL REQUIREMENTS.
(Revision of Table I, B 107 - 55 T)

Alloy	Zinc, per cent	Silicon, max, per cent	Calcium, max, per cent	Other Impurities, max, per cent
AZ31B	0.6 to 1.4	0.30	...	0.30
AZ61A	0.40 to 1.5	0.30	...	0.30
AZ80A	0.20 to 0.8	0.30	...	0.30
M1A	...	0.30	0.30	0.30
ZK60A	4.8 to 6.2	...	...	0.30

New Tables.—Add new Tables VII, VIII and IX as shown, renumbering present Tables VII and VIII as Tables X, and XI.

TABLE VII.—LENGTH TOLERANCES.
(New Table VII, B 107 - 55 T)

Circumscribing Circle Diameter (Shapes); Specified Diameter (Rod); Specified Width or Depth, Whichever Greater (Bar), in.	Allowable Deviation from Specified Length, in, for Specified Length Given in Feet			
	12 and under	Over 12 to 30, incl	Over 30 to 50, incl	Over 50
Under 3.000	+1/8	+1/4	+3/8	+1
3.000 to 7.999	+3/16	+5/16	+7/16	+1
8.000 and over	+1/4	+3/8	+1/2	+1

TABLE VIII.—SURFACE ROUGHNESS.
(New Table VIII, B 107 - 55 T)

Specified Section Thickness, in.	Allowable Depth of Defect max in.[a]
Under 0.064	0.0015
0.064 to 0.125	0.002
0.126 to 0.188	0.0025
0.189 to 0.250	0.003
0.251 to 0.500	0.004
0.501 and over	0.008

[a] Defects include die lines and handling marks.

TABLE IX.—CORNER AND FILLET RADII.
(New Table IX, B 107 - 55 T)

Specified Radius, in.	Allowable Deviation from Specified Radius, in.
Sharp corners	+1/64
Under 0.188	±1/64
0.188 and over	±10 per cent

Tentative Specification for Aluminum-Base Alloy Permanent Mold Castings (B 108 - 55 T):

Table I.—For alloy GS42A add a limit of 0.15 per cent max for other elements, total. For alloy GZ42A add a limit of 0.15 per cent max for other elements, total. For alloy ZC81B revise the silicon limit from "0.35" to read "0.25" per cent max, and the limit for other elements, each, from "0.15" to read "0.10" per cent max. Add alloy SC103A with the following chemical requirements:

Aluminum, per cent	remainder
Copper, per cent	2.0 to 4.0
Iron, per cent	1.2
Silicon, per cent	8.5 to 10.5
Manganese, per cent	0.50
Magnesium, per cent	0.50 to 1.5
Zinc, per cent	1.0
Titanium, per cent	0.25
Nickel, per cent	0.50
Other elements, total, per cent	0.50

Delete Footnote *a* and the table of former alloy designations and reletter subsequent footnotes accordingly.

Table II.—Add alloy SC103A with the following tensile requirements:

Condition	T5 (aged)
Tensile Strength, min, psi.	31 000
Elongation in 2 in., min, per cent	[c]

Table IV.—Add alloy SC103A with the following properties and characteristics:

Approximate Melting Range, deg Fahr	970 to 1080
Foundry Characteristics	
Resistance to Hot Cracking	1
Pressures Tightness	2
Fluidity	1
Solidification Shrinkage Tendency	2
Normally Heat Treated	aged only
Other Characteristics	
Resistance to Corrosion	3
Machining	3
Polishing	4
Electroplating	3
Anodizing (Appearance)	5
Chemical Oxide Coating (Protection)	3
Strength at Elevated Temperature	3
Suitability for Welding	2
Suitable for Brazing	No

Tentative Specifications for Aluminum-Alloy Sheet and Plate for Pressure Vessel Applications (B 178 – 55 T):

Table I.—For alloy MG11A, revise the figures under the column headed "Copper, per cent" from "0.20" to read "0.25"; and revise the figures under the column headed "Zinc, per cent" from "0.10" to read "0.25."

Table II.—Revise values for alloy GM40A for -O, -H32, -H34. -H36, and -H112 tempers as shown in the accompanying Table X, retaining present Footnote *b* referenced after the tensile strength values for the "O" temper.

TABLE X.—TENSILE REQUIREMENTS.
(Revisions in Table II, B 178 – 55 T)

Temper	Thickness, in.	Tensile Strength, min, psi	Yield Strength, min, psi	Elongation in 2 in., min, per cent
O	0.020 to 0.050	35 000	14 000	15
	0.051 to 0.249	35 000	14 000	18
	0.250 to 2.000	35 000	14 000	14
H32	0.020 to 0.050	40 000	28 000	6
	0.051 to 0.249	40 000	28 000	8
	0.250 to 2.000	40 000	28 000	12
H34	0.020 to 0.050	44 000	34 000	5
	0.051 to 0.249	44 000	34 000	6
	0.250 to 1.000	44 000	34 000	10
H36	0.020 to 0.050	47 000	38 000	4
	0.051 to 0.162	47 000	38 000	6
H112	0.250 to 0.500	36 000	18 000	8
	0.501 to 1.000	35 000	16 000	10
	1.001 to 2.000	35 000	14 000	14
	2.001 to 3.000	34 000	14 000	14

New Section.—Add a new Section 15 titled "Marking for Identification" and renumber the succeeding sections. The new Section 15 shall read:

15. (*a*) When identification marking is specified on the purchase order, all sheet and plate shall be marked with the manufacturer's name or trade mark and the applicable alloy and temper designations, the latter to be in accordance with the ASTM codification systems (B 275 and B 296) or the commercial systems. Identification characters shall have a minimum height of $\frac{1}{4}$ in. The marking material shall be such as to resist obliteration during normal handling and shall be removable by normal cleaning methods; however, ghost images of the characters may remain. On flat sheet and plate, markings shall appear at each end. Coils shall be marked near the outside end.

(*b*) The foregoing requirements are minimum; marking systems which involve added information, larger characters, and greater frequencies are acceptable under this specification.

Tentative Specifications for Magnesium-Base Alloy Permanent Mold Castings (B 199 – 55 T):

Section 2.—Renumber Item (*4*) as (*7*) and add the following:

(*4*) Casting drawing giving all necessary dimensions and showing amount of finish left for machining.

(*5*) Surface treatment (Section 12).

(*6*) Special inspection requirements (Section 13(*c*)).

Section 12.—Revise to read as follows:

12.—The castings shall be of uniform quality and condition, free of cracks or other injurious defects, and shall be well cleaned before inspection

Unless otherwise specified, the castings shall be chrome pickled prior to shipment. See Explanatory Note 4.

Section 13.—Add a new Paragraph (*c*) as follows:

(*c*) Special inspection requirements such as pressure testing, X-ray, fluorescent penetrant, etc., must be stated on the order. Acceptance standards under special inspection requirements shall be as agreed upon by the purchaser and the manufacturer.

Explanatory Note.—Add a new Note 4 as follows:

NOTE 4.—The chrome pickle affords measurable protection against corrosion and tarnish during shipment and storage of the castings. After pickling, the castings will be grey to bronze or yellow in color depending on alloy and condition. The chrome pickle is not recommended for castings containing laminated inserts. Such castings should be ordered shipped without surface treatment or protected with a slushing compound.

Table I.—Delete Footnote *a*, renumbering *b* as *a* and revise columns relating to zinc, silicon and other impurities as follows:

Alloy[a]	Zinc, per cent	Silicon, max, per cent	Other Impurities, max, per cent
AM100A	0.30 max	0.30	0.30
AZ92A......	1.6 to 2.4	0.30	0.30

Table II.—Delete Footnote *a* and renumber Footnote *b* as *a*.

Tentative Specifications for Aluminum-Alloy Sheet and Plate (B 209 – 55 T):

Table I.—For alloy MG11A, revise the figures under the column headed "Copper, Per Cent" from "0.20" to read "0.25;" and revise the figures under the column headed "Zinc, Per Cent" from "0.10" to read "0.25."

Table II.—Revise values for Alloy GM40A for -O, -H32, -H34, -H36, and -H112 tempers as shown in the accompanying Table XI.

TABLE XI.—TENSILE REQUIREMENTS.
(Revisions in Table II, B 209 – 55 T)

Temper	Thickness, in.	Tensile Strength, min, psi	Yield Strength, min, psi	Elongation in 2 in., min, per cent
O................	0.020 to 0.050..............	35 000	14 000	15
	0.051 to 0.249..............	35 000	14 000	18
	0.250 to 2.000..............	35 000	14 000	14
H32..............	0.020 to 0.050..............	40 000	28 000	6
	0.051 to 0.249..............	40 000	28 000	8
	0.250 to 2.000..............	40 000	28 000	12
H34..............	0.020 to 0.050..............	44 000	34 000	5
	0.051 to 0.249..............	44 000	34 000	6
	0.250 to 1.000..............	44 000	34 000	10
H36..............	0.020 to 0.050..............	47 000	38 000	4
	0.051 to 0.162..............	47 000	38 000	6
H112.............	0.250 to 0.500..............	36 000	18 000	8
	0.501 to 1.000..............	35 000	16 000	10
	1.001 to 2.000..............	35 000	14 000	14
	2.001 to 3.000..............	34 000	14 000	14

New Section.—Add a new Section 15 titled "Marking for Identification" to read as follows, renumbering subsequent sections accordingly:

15. (*a*) When identification marking is specified on the purchase order, all sheet and plate shall be marked with the manufacturer's name or trade mark and the applicable alloy and temper designations, the latter to be in accordance with the ASTM codification systems (ASTM Designations: B 275 and B 296) or the commercial systems. Identification characters shall have a minimum height of $\frac{1}{4}$ in. The mark-

ing material shall be such as to resist obliteration during normal handling and shall be removable by normal cleaning methods; however, ghost images of the characters may remain. On flat sheet and plate, markings shall appear at each end. Coils shall be marked near the outside end.

(*b*) The foregoing requirements are minimum; marking systems which involve added information, larger characters and greater frequencies are acceptable under these specifications.

Tentative Specifications for Aluminum-Alloy Drawn Seamless Tubes (B 210 – 55 T):

Table I.—For alloy MG11A, revise the figures under the column headed "Copper, Per Cent" from "0.20" to read "0.25;" and revise the figures under the column headed "Zinc, Per Cent" from "0.10" to read "0.25."

New Section.—Add a new Section 14 titled "Marking for Identification" to read as follows, renumbering all subsequent sections accordingly:

14. (*a*) When identification marking is specified on the purchase order, all tubes in straightened lengths $\frac{3}{8}$ in. in diameter or across flats and larger shall be marked with the manufacturer's name or trade mark and the applicable alloy and temper designations, the latter to be in accordance with the ASTM codification systems (ASTM Designations: B 275 and B 296) or the commercial systems. The identification characters shall have a minimum height of $\frac{1}{8}$ in. The marking material shall be such as to resist obliteration during normal handling and shall be removable by normal cleaning methods; however, ghost images of the characters may remain. Markings shall appear at each end of each piece. Straightened material smaller than $\frac{3}{8}$ in. diameter or across flats and all material supplied in coils shall be either tagged (in bundles if desired) or marked as individual pieces with the above information.

(*b*) The foregoing requirements are minimum; marking systems which involve added information, larger characters, and greater frequencies are acceptable under these specifications.

Tentative Specifications for Aluminum-Alloy Bars, Rods and Wire (B 211 – 55 T):

New Section.—Add a new Section 14 titled "Marking for Identification" to read as follows, renumbering all subsequent sections accordingly:

14. (*a*) When identification marking is specified on the purchase order, all bars, rods (shapes) and wire in straightened lengths $\frac{3}{8}$ in. in diameter or across flats and larger shall be marked with the manufacturer's name or trade mark and the applicable alloy and temper designations, the latter to be in accordance with the ASTM codification systems (ASTM Designations: B 275 and B 296) or the commercial systems. The identification characters shall have a minimum height of $\frac{1}{8}$ in. The marking material shall be such as to resist obliteration during normal handling and shall be removable by normal cleaning methods; however, ghost images of the characters may remain. Markings shall appear at each end of each piece. Straightened material smaller than $\frac{3}{8}$ in. diameter or across flats and all material supplied in coils shall be either tagged (in bundles, if desired) or marked as individual pieces with the above information.

(*b*) The foregoing requirements are minimum; marking systems which involve added information, larger characters, and greater frequencies are acceptable under these specifications.

Tentative Specifications for Magnesium-Base Alloy Extruded Tubes (B 217 – 55 T):

Section 2.—Change to read as follows:

(*1*) Quantity in pieces, pounds or length,
(*2*) Alloy (Section 4, Table I),
(*3*) Condition (Section 7, Table II),
(*4*) Outside diameter or inside diameter and wall thickness,
(*5*) Length per piece. (Table VIII),
(*6*) Surface treatment (Section 13), and
(*7*) Place of inspection (Section 14 (*a*)).

Section 12.—Delete Paragraph (*b*) and add the following:

(*b*) Straightness shall conform to the requirements of Table VII and Table XI.

(*c*) Variations in length shall not exceed the amount permitted by Table VIII.

(*d*) Roughness of surface shall be not greater than permitted by Table IX.

(*e*) Deviation from specified radius shall not exceed that permitted by Table X.

(*f*) Angles, curved surfaces, flat surfaces, and squareness of cut ends shall conform to the following:

(*1*) *Angles.*—Allowable deviation from specified angle, ± 2 deg.

(*2*) *Curved Surfaces.*—Allowable deviation from specified contour, 0.005 in. per in. of chord length, 0.005 in., min; not applicable to more than 90 deg or any arc.

(*3*) *Flat Surfaces.*—Allowable deviation from flat, 0.004 in. per in. of width, 0.004 in., min. Not applicable to surfaces of walls 0.250 in. or less in thickness which completely enclose a void.

(*4*) *Cut Ends.*—Allowable deviation from square, 1 deg.

Section 13.—Add a new paragraph to read as follows:

Unless otherwise specified, the extruded tubing shall be oiled or chrome pickled, as elected by the purchaser, prior to shipment. See Explanatory Note 2.

TABLE XII.—CHEMICAL REQUIREMENTS.
(Revision of Table I, B 217 – 55 T)

Alloy[a]	Manganese, min, per cent	Zinc, per cent	Silicon, max, per cent	Calcium, max, per cent	Other Elements, max, per cent
AZ31B	0.20	0.6 to 1.4	0.30	...	0.30
AZ61A	0.15	0.40 to 1.5	0.30	...	0.30
M1A	1.2	...	0.30	0.30	0.30
ZK60A	...	4.8 to 6.2	...	...	0.30

TABLE XIII.—STRAIGHTNESS.
(New Table VII, B 217 – 55 T)

Specified Outside Diameter; Width or Depth (Whichever Greater), in.	Allowable Deviation from Straight, in.[a]	
	In Each Foot of Length	In Total Length of Piece
0.500 to 5.999	0.010	0.010 × length (ft)
6.000 and over	0.020	0.020 × length (ft)

[a] For all sizes of other than round tube, tolerance is applicable when weight of tube on flat surface minimizes deviation.

Explanatory Notes.—Change "Note" in heading to "Notes" and change "Note" to "Note 1." Add a Note 2 as follows:

NOTE 2.—Either the chrome pickle or the oil finish affords a measurable protection against tarnish and corrosion during shipment and storage of magnesium tubing.

Table I.—Revise the columns indicated in the accompanying Table XII, leaving other columns and note unchanged.

New Tables VII, VIII, IX, X, and XI.—Add the accompanying Tables XIII, XIV, XV, XVI and XVII of tolerances on length, twist, straightness, surface roughness, and corner radii, and renumber the present Table VII as Table XII.

TABLE XIV.—LENGTH.
(New Table VIII, B 217 – 55 T)

Specified Outside Diameter, Width or Depth (Whichever Greater), in.	Allowable Deviation from Specified Length, in., for Specified Length, Given in Feet			
	12 and under	Over 12 to 30, incl	Over 30 to 50, incl	Over 50
Under 0.500	...	...	...	...
0.500 to 1.249	+1/8	+1/4	+3/8	+1
1.250 to 2.999	+1/8	+1/4	+3/8	+1
3.000 to 7.999	+3/16	+5/16	+7/16	+1
8.000 and over	+1/4	+3/8	+1/2	+1

TABLE XV.—SURFACE ROUGHNESS.
(New Table IX, B 217 – 55 T)

Specified Section Thickness, in.	Allowable Depth of Defect[a], max, in.
Under 0.064	0.0015
0.064 to 0.125	0.002
0.126 to 0.188	0.0025
0.189 to 0.250	0.003
0.251 to 0.500	0.004
0.501 and over	0.008

[a] Defects include die lines and handling marks.

Tentative Specifications for Aluminum-Alloy Extruded Bars, Rods, and Shapes (B 221 – 55 T):

New Section.—Add new Section 14 titled "Marking for Identification" to read as follows, renumbering all subsequent sections accordingly:

14. (*a*) When identification marking is specified on the purchase order, all bars, rods, and shapes 3/8 in. in diameter or across flats and larger shall be marked with the manufacturer's name or trade mark and the applicable alloy and

temper designations, the latter to be in accordance with the ASTM codification systems (ASTM Designations: B 275 and B 296) or the commercial systems. Identification characters shall have a minimum height of ⅛ in. The marking material shall be such as to resist obliteration during normal handling and shall be removable by normal cleaning methods; however, ghost images of the characters may remain. Markings shall appear at each end of each piece. Material smaller than ⅜-in. diameter or across flats shall be either tagged (in bundles, if desired) or marked as individual pieces with the above information.

(*b*) The foregoing requirements are minimum; marking systems which involve added information, larger characters, and greater frequencies are acceptable under these specifications.

TABLE XVI.—CORNER RADII.
(New Table, X, B 217 - 55 T)

Specified Radius, in.	Allowable Deviation from Specified Radius, in.
Sharp corners	+1/64
Under 0.188	±1/64
0.188 and over	±10 per cent

TABLE XVII.—TWIST.
(New Table XI, B 217 - 55 T)

Specified Width or Depth (Whichever Greater), in.	Allowable Deviation from Straight, deg.	
	In Each Foot of Length	In Total Length of Piece
Under 1.500	1	Length (ft) × 1
1.500 to 2.999	½	Length (ft) × ½, not over 5
3.000 and over	¼	Length (ft) × ¼, not over 3

Tentative Specifications for Aluminum-Alloy Drawn Seamless Tubes for Condensers and Heat Exchangers (B 234 - 55 T):

Section 5 (*a*).—Revise the first sentence to read as follows:

Samples for chemical analysis of type M1A, GS11A, and GS11C shall be taken in accordance with the Method of Sampling Wrought Non-Ferrous Metals and Alloys for Determination of Chemical Composition (ASTM Designation: E 55), except that the weight of the prepared sample may be a minimum of 75 g.

Section 15.—Revise to read as follows:

15. (*a*) *Diameter*.—The outside diameter of the tubes shall not vary from that specified by more than the amounts as measured by "go" and "no-go" ring gages prescribed in Table III.

(*b*) *Wall Thickness*.—No tube at its thinnest point shall be less than the thickness specified. The mean wall thickness and the wall thickness at any point shall not exceed that specified by more than the amount prescribed in Table IV.

(*c*) *Length*—The length shall be not less than that specified, when measured at a temperature of 20 C (68 F), but may be more than that specified by the amount prescribed in Table V.

(*d*) *Straightness*.—The tubes shall not vary from straight by more than the amounts prescribed in Table VI.

New Section.—Add a new Section 17 titled "Marking for Identification," re-

TABLE XVIII.—PERMISSIBLE VARIATIONS IN DIAMETER.
(Revision of Table III, B 234 - 55 T)

Specified Diameter, in.	Permissible Variation in Diameter plus or minus, in.
0.500 and under	0.003
0.501 and 1.000 incl.	0.004
1.001 to 2.000 incl.	0.005

TABLE XIX.—PERMISSIBLE VARIATIONS IN WALL THICKNESS.
(Revision of Table IV, B 234 - 55 T)

Specified Wall Thickness,[a] in.	Permissible Variation in Mean[b] Wall Thickness, plus only, in.	Permissible Variation in Wall Thickness at any Point, plus only, in.
0.035	0.004	0.006
0.049	0.006	0.009
0.058	0.007	0.010
0.065	0.008	0.012
0.072	0.009	0.013
0.083	0.010	0.015
0.095	0.011	0.017
0.109	0.013	0.020
0.120	0.014	0.022
0.134	0.016	0.024
0.145	0.017	0.026
0.154	0.018	0.028
0.179	0.021	0.032
0.200	0.024	0.036

[a] If specified wall thickness is not shown, the tolerance for the next thicker wall shall be used.

[b] Mean wall thickness is the average of two measurements taken opposite each other.

numbering all subsequent sections accordingly:

17. (*a*) When identification marking is specified on the purchase order, all tubes $\frac{3}{8}$ in. in diameter and larger shall be marked with the manufacturer's name or trade mark and the applicable alloy and temper designations, the latter to be in accordance with the ASTM codification systems (ASTM Designations: B 275 and B 296) or the commercial systems. The identification characters shall have a minimum height of $\frac{1}{8}$ in. The marking material shall be such as to resist obliteration during normal handling and shall be removable by normal cleaning methods; however, ghost images of the characters may remain. Markings shall appear at each end of each piece. Material smaller than $\frac{3}{8}$-in. diameter shall be either tagged (in bundles, if desired) or marked as individual pieces with the above information.

(*b*) The foregoing requirements are minimum; marking systems which involve added information, larger characters, and greater frequencies are acceptable under these specifications.

Tables III, IV, and V.—Revise to read as shown in the accompanying Tables XVIII, XIX, and XX.

New Table.—Add a new Table VI to read as shown in the accompanying Table XXI.

TABLE XX.—PERMISSIBLE VARIATIONS IN LENGTH.
(Revision of Table V, B 234 – 55 T)

Specified Length, ft	Permissible Variation in Length, plus only, in.
16 and under	$\frac{1}{8}$
Over 16 to 30, incl	$\frac{1}{4}$
Over 30 to 50, incl	$\frac{3}{8}$
Over 50	1

TABLE XXI.—PERMISSIBLE VARIATIONS FROM STRAIGHT.
(New Table VI, B 234 – 55 T)

Specified Diameter, in.	Permissible Variation in Each Foot of Length, in.	Permissible Variation in Total Length of Piece, in.
0.375 to 5.999, incl	0.010	0.010 × length (ft)

Tentative Specifications for Aluminum-Alloy Extruded Tubes (B 235 – 55 T):

New Section.—Add a new Section 14 titled "Marking for Identification" to read as follows, renumbering all subsequent sections accordingly:

14. (*a*) When identification marking is specified on the purchase order, all tubes shall be marked with the manufacturer's name or trade mark and the applicable alloy and temper designations, the latter to be in accordance with the ASTM codification systems (ASTM Designation B 275 and B 296) or the commercial systems. The identification characters shall have a minimum height of $\frac{1}{8}$ in. The marking material shall be such as to resist obliteration during normal handling and shall be removable by normal cleaning methods; however, ghost images of the characters may remain. Markings shall appear at each end of each piece.

(*b*) The foregoing requirements are minimum; marking systems which involve added information, larger characters, and greater frequencies are acceptable under these specifications.

Tentative Specifications for Aluminum Bars for Electrical Purposes (Bus Bar) (B 236 – 55 T):

New Section.—Add a new Section 17 titled "Marking for Identification" to read as follows, renumbering all subsequent sections accordingly:

17. (*a*) When identification marking is specified on the purchase order, all bars shall be marked with the manufacturer's name or trade mark and the applicable alloy and temper designations, the latter to be in accordance with the ASTM codification systems (ASTM Designations: B 275 and B 296) or the commercial systems. Identification characters shall have a minimum height of $\frac{1}{4}$ in. The marking material shall be such as to resist obliteration during normal handling and shall be removable by normal cleaning methods; however, ghost images of the characters may remain. Markings shall appear at each end of each piece.

(*b*) The foregoing requirements are minimum; marking systems which involve added information, larger characters, and greater frequencies are acceptable under these specifications.

Tentative Specifications for Aluminum-Alloy Pipe (B 241 – 55 T):

New Section.—Add a new Section 14 titled "Marking for Identification" to read as follows, renumbering all subsequent sections accordingly:

14. (*a*) When identification marking is specified on the purchase order, all pipe shall be marked with the manufacturer's name or trade mark and the applicable alloy and temper designations, the latter to be in accordance with the ASTM codification systems (ASTM Designations: B 275 and B 296) or the commercial systems. The identification characters shall have a minimum height of $\frac{1}{8}$ in. The marking material shall be such as to resist obliteration during normal handling and shall be removable by normal cleaning methods; however, ghost images of the characters may remain. Markings shall appear at each end of each piece.

(*b*) The foregoing requirements are minimum; marking systems which involve added information, larger characters, and greater frequencies are acceptable under these specifications.

Tentative Specifications for Aluminum Alloy Bars, Rods, and Shapes for Pressure Vessel Application (B 273 – 55 T):

New Section.—Add a new Section 14 titled "Marking for Identification" to read as follows, renumbering all subsequent sections accordingly:

14. (*a*) When identification marking is specified on the purchase order, all bars, rods, and shapes $\frac{3}{8}$ in. in diameter or across flats and larger shall be marked with the manufacturer's name or trade mark and the applicable alloy and temper designations, the latter to be in accordance with the ASTM codification systems (ASTM Designations: B 275 and B 296) or the commercial systems. Identification characters shall have a minimum height of $\frac{1}{8}$ in. The marking material shall be such as to resist obliteration during normal handling and shall be removable by normal cleaning methods; however, ghost images of the characters may remain. Markings shall appear at each end of each piece. Material smaller than $\frac{3}{8}$-in. diameter or across flats shall be either tagged (in bundles, if desired) or marked as individual pieces with the above information.

(*b*) The foregoing requirements are minimum; marking systems which involve added information, larger characters, and greater frequencies are acceptable under these specifications.

Tentative Specifications for Aluminum-Alloy Pipe and Tube for Pressure Vessel Applications (B 274 – 55 T):

Table I.—For alloy MG11A revise the figures under the column headed "Zinc, per cent" from "0.10" to read "0.25;" and under the column headed "Copper, per cent" from "0.20" to read "0.25."

Section 13.—Revise Paragraph (*b*) to read:

(*b*) *Wall Thickness.*—For schedules other than 5S and 10S the wall thickness at any point shall be not more than 12.5 per cent under the nominal thickness shown in Table III. For schedules 5S and 10S the wall thickness at any point shall not exceed nor be less than 12½ percent of the nominal wall thickness as given in Table III.

Revise Paragraph (*c*) to read:

(*c*) *Weight.*—For schedules other than 5S and 10S the weight of the pipe shall not exceed the theoretical weight given in Table III by more than 8 per cent. For schedules 5S and 10S a weight tolerance does not apply, and the weight will vary in accordance with the wall thickness as prescribed in Paragraph (*b*).

New Section.—Add a new Section 16 titled "Marking for Identification" to read as follows, renumbering all subsequent sections accordingly:

16. (*a*) When identification marking is specified on the purchase order, all tubes and pipes shall be marked with the manufacturer's name or trade mark and the applicable alloy and temper designations, the latter to be in accordance with the ASTM codification systems (ASTM Designations: B 275 and B 296) or the commercial systems. The identification characters shall have a minimum height of $\frac{1}{8}$ in. The marking material shall be such as to resist obliteration during normal handling and shall be removable by normal cleaning methods; however, ghost images of the characters may remain. Markings shall appear at each end of each piece.

(*b*) The foregoing requirements are minimum; marking systems which involve added information, larger characters, and greater frequencies are acceptable under these specifications.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following two tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Method of:

Measuring Thickness of Anodic Coatings on Aluminum by Means of the Filmeter (**B 244 - 49 T**).

Tentative Recommended Practice for:

Temper Designation of Light Metals and Alloys, Cast and Wrought (**B 296 - 54 T**).

TABLE XXII.—CHEMICAL REQUIREMENTS.
(Revision of Table I, B 179 - 55)

	ZC81A	ZC81B
Aluminum, per cent	remainder	remainder
Copper, per cent	0.40 to 1.0	0.40 to 1.0
Iron, per cent	0.8	1.1
Silicon, per cent	0.25	0.25
Manganese, per cent	0.6	0.6
Magnesium, per cent	0.25 to 0.50	0.25 to 0.50
Zinc, per cent	7.0 to 8.0	7.0 to 8.0
Chromium, per cent	0.35	0.35
Titanium, per cent	0.25	0.25
Nickel, per cent	0.15	0.15
Other elements:		
Each, per cent	0.10	0.10
Total, per cent	0.25	0.25

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends that the following two standard specifications be revised for immediate adoption as indicated below, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Specifications for Magnesium Ingot and Stick for Remelting (B 92 - 55):

Section 1.—Delete note from Section 1.

Section 2.—Revise to read as follows:

(*1*) Quantity, in pieces or pounds.

(*2*) Form; magnesium ingot and stick are normally furnished in the following commercial forms unless otherwise specified in the contract:

Form	Approximate Size and Shape, in.		Approximate Weight, lb
Ingot (3 notches)	4 by 4½ by 28		20.0
Interlocking, self-palletizing ingot	8¼ by 2¾ by 33		42.0
	Diameter	Length	
Stick	0.65	12	0.25
	0.92	12	0.50
	1.3	12	1.0
	1.3	24	2.0

TABLE XXIII.—CHEMICAL REQUIREMENTS.
(Addition to Table I, B 179 - 55)

	SC103A	SC114A
Aluminum, per cent	remainder	remainder
Copper, per cent	2.0 to 4.0	3.0 to 4.5
Iron, per cent	1.0	1.0
Silicon, per cent	8.5 to 10.5	10.5 to 12.0
Manganese, per cent	0.50	0.50
Magnesium, per cent	0.6 to 1.5	0.10
Zinc, per cent	1.0	0.9
Titanium, per cent	0.25	...
Nickel, per cent	0.50	0.50
Tin, per cent	...	0.35
Other elements, Total, per cent	0.50	0.50

Standard Specification for Aluminum-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 179 - 55):

Table I.—For alloy CS66A revise the magnesium range from "0.25 to 0.40" to read "0.25 to 0.6" per cent. For alloy G4A revise the iron limit from "0.45" to read "0.40" per cent, max. For alloy SN122A revise the iron limit from "1.0" to read "1.1" per cent, max, and the magnesium range from "0.9 to 1.3" to read "0.8 to 1.3" per cent. For alloy ZC60A revise the iron limit from "1.3"

to read "1.2" per cent, max, and the magnesium range from "0.25 to 0.45" to read "0.30 to 0.45" per cent. For alloy ZG32A revise the magnesium range from "1.4 to 1.8" to read "1.5 to 1.8" per cent. For alloy ZG42A revise the magnesium range from "1.8 to 2.4" to "1.9 to 2.4" per cent. For alloy ZG61B revise the iron limit from "0.45" to read "0.40" per cent max, and the magnesium range from "0.6 to 0.8" to read "0.65 to 0.8" per cent. Delete alloy ZC81A–B and replace it with ZC81A and ZC81B with the chemical requirements shown in the accompanying Table XXII. Add alloys SC103A and SC114A with the chemical requirement shown in the accompanying Table XXIII.

Delete Footnote *a* and the table of former alloy designations and reletter subsequent footnotes accordingly.

WITHDRAWAL OF STANDARD

The committee recommends withdrawal of the following standard:

Standard Specification for Aluminum Ingots for Remelting (B 24 – 46).

REPORT OF COMMITTEE B-8

ON

ELECTRODEPOSITED METALLIC COATINGS*

Committee B-8 on Electrodeposited Metallic Coatings held two meetings during the year: on September 22, 1955, at ASTM Headquarters, Philadelphia, Pa., and in Buffalo, N. Y., on March 1, 1956.

The membership of the committee now consists of 128 members, of whom 54 are classified as producer, 18 as consumer, 56 as general interest, and 16 as consulting members.

George Best has been appointed to represent the committee on Committee D-19 on Industrial Water, and A. H. DuRose has been appointed to Subcommittee 16 on Thickness Measurement of Committee E-1.

The committee has approved by letter ballot the submission of its specifications and recommended practices to the American Standards Association for approval as American Standards.

The officers elected for the ensuing term of two years are as follows:

Chairman, C. H. Sample.

Vice-Chairman, A. B. Saltonstall.

Secretary, D. M. Biggs.

Advisory Committee Member-at-Large, R. E. Harr.

EDITORIAL CHANGE

The committee recommends the following editorial change in the Standard Specifications for Electrodeposited Coatings of Nickel and Chromium on Copper and Copper-Base Alloys (**B 141 - 55**):[1]

Section 2.—Add a footnote reference to the end of this section, the footnote to read as follows: "Nickel deposited by chemical reduction and otherwise meeting the requirements of the particular application may be accepted under these specifications."

This footnote is added in the interest of uniformity, since it is identical to the one now appearing in the Specifications for Electrodeposited Coatings of Nickel and Chromium on Steel (A 166).

The recommendation in this report has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Papers, Specifications, and Definitions (F. Ogburn, chairman).—The Symposium Committee, under the chairmanship of W. L. Pinner, presented a highly successful one-day Symposium on Properties, Tests and Performance of Electrodeposited Metallic Coatings,[3] on February 29, 1956, at Committee Week in Buffalo. This symposium will be repeated at the Second ASTM Pacific Area National Meeting in Los Angeles, Calif. in September, 1956.

A new Section C on Nickel and Nickel Alloys Deposited by Chemical Reduction was established under the chairmanship of Walter Moline.

The appointment of a new task group

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17-22, 1956.

[1] 1955 Book of ASTM Standards, Part 2.

[2] The letter ballot vote on this recommendation was favorable; the results of the vote are on record at ASTM Headquarters.

[3] Issued as separate publication *ASTM No. 197.*

has been authorized to consider the matter of marking electroplated items to indicate compliance with ASTM specifications.

Subcommittee II on Performance Tests (W. L. Pinner, chairman).—Inspection and rating of steel panels plated with copper-nickel-chromium, and nickel-chromium are continuing. Additional panels, designated as Program 4, were exposed during March and April, 1956. Panels exposed during 1955 for the purpose of obtaining typical corrosion patterns and demonstrating rating numbers are being removed and preserved in various stages of deterioration.

Inspections and ratings of zinc-plated steel panels, many with conversion coatings, are being continued. Massive steel panels, cadmium plated, with conversion coatings, are ready to be exposed.

A modification of present rating systems has been proposed and it will be used at the May inspection.

New task groups have been appointed on white brass coatings for indoor exposure, under the chairmanship of H. Kahler, and on outdoor exposure of copper-nickel-chromium coatings on aluminum, under the chairmanship of J. W. Kerstetter.

Subcommittee III on Conformance Tests (R. F. Ledford, chairman).—A task group under the chairmanship of C. F. Waite has not found it advisable to include in the Method of Test for Local Thickness of Electrodeposited Coatings (A 219) descriptions and procedures for the anodic solution method and the jet method for measuring thickness.

A task group under the chairmanship of W. Moline is reviewing methods for measuring thickness of chromium deposits.

The task group under M. Frager on effect of temperature on service life of electrodeposited and hot-dipped zinc coatings has recommended the addition of a precautionary appendix to the Specifications for Electrodeposited Coatings of Zinc on Steel (A 164).

Subcommittee IV on Electroplating Practice (Max Frager, chairman).—A Recommended Practice for the Preparation of Lead and Lead Alloys for Electroplating has been prepared and is ready for letter ballot of Committee B-8.

A newly appointed task group under the chairmanship of H. Kafarski is investigating the feasibility of preparing a recommended practice on the analysis and treatment of water for use in plating and allied operations.

Subcommittee V on Supplementary Treatments for Electrodeposited Metallic Coatings (Marc Darrin, chairman) is directly involved in the exposure tests of the steel panels, zinc and cadmium plated with conversion coatings, mentioned above under the activities of Subcommittee II.

Section F on supplemental organic coatings has acquired some interesting information regarding treatment of chromium plate prior to application of clear organic coatings. The section has been asked to develop information of this type with respect to organic coatings over chromated zinc plate.

This report has been submitted to letter ballot of the committee, which consists of 128 voting members; 115 members returned their ballots, of whom 107 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

C. H. SAMPLE,
Chairman.

R. B. SALTONSTALL,
Secretary.

REPORT OF COMMITTEE B-9

ON

METAL POWDERS AND METAL POWDER PRODUCTS*

Committee B-9 on Metal Powders and Metal Powder Products held one meeting during the year: in New York City on February 22, 1956.

The officers and members of the Advisory Committee elected for the ensuing term of two years are as follows:

Chairman, J. L. Bonanno.
Vice-Chairman, H. R. Biehl.
Secretary, C. G. Johnson.
Advisory Committee: F. V. Lenel, M. H. Meighan, W. M. Shafer, and G. L. Werley.

NEW TENTATIVES

The committee recommends that the following be accepted for publication as tentative as appended hereto:[1]

Specifications for:

High-Density Iron, Sintered Metal Powder Structural Parts, and
Low- and Medium-Density Iron, Sintered Metal Powder Structural Parts.

Methods of:

Test for Green Strength of Compacted Metal Powder Specimens, and
Test for Density of Cemented Carbides.

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption the following revision of the Standard Method of Test for Sieve Analysis of Granular Metal Powders (**B 214 – 48**),[2] and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revision may be referred to letter ballot of the Society:

Section 2 (*b*).—Change to read as follows:

(*b*) *Sieve Shaker.*—A mechanically operated sieve shaker, which imparts to the set of sieves a rotary motion and tapping action of uniform speed, shall be used. The number of taps per minute shall be between 140 and 160. The sieve shaker shall be fitted with a hard maple plug to receive the impact of the tapping device. The entire apparatus shall be rigidly mounted by bolting to a solid foundation, preferably of concrete. Preferably a time switch shall be provided to insure accuracy of duration of the test.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following tentatives be continued without revision in view of pending changes:

Tentative Specifications for:

Metal Powder Sintered Bearings (Oil Impregnated) (B 202 – 55 T),
Sintered Metal Powder Structural Parts from Bronze (B 255 – 54 T), and
Sintered Metal Powder Structural Parts from Brass (B 282 – 53 T).

Tentative Method of:

Subsieve Analysis of Granular Metal Powders by Air Classification (B 293 – 54 T).

Tentative Recommended Practice for:

Hardness Testing of Cemented Carbides (B 294 – 54 T).

The recommendations in this report have been submitted to letter ballot of

the committee, the results of which will be reported at the Annual Meeting.[3]

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] These tentatives appear in the 1956 Supplement to Book of ASTM Standards, Part 2.

[2] 1955 Book of ASTM Standards, Part 2.

ACTIVITIES OF SUBCOMMITTEES

The seven active subcommittees and sections of the committee met twice during the year: in Philadelphia, Pa., on October 18 to 19, 1955, and prior to the main committee meeting in New York City on February 21 to 22, 1956.

Subcommittee I on Nomenclature and Technical Data (F. N. Rhines, chairman) formulated the following scope of activities to guide its efforts:

> To determine the technical consequences of producing materials, or otherwise operating within specifications written by the several subcommittees, and to discover, if possible, the causes of any unexpected and unwanted results when working within these specifications. This is to be accomplished, insofar as may be feasible, by gathering data from those who are producing the specified materials, or are using the specified methods, as will best reveal the effects of using the specifications. The data so gathered are to be analyzed and the results made available in suitable form.

It is expected that the results will reveal flaws, if any, in the operation of the specifications and perhaps indicate how these could be corrected.

The program of gathering data relative to the operation of specification B 222 – 52 is continuing under the leadership of J. M. Copeland. Efforts to gather data on the *K*-factor for bearings manufactured under the restrictions of Specifications B 202 – 55 T have been suspended owing to a decision in Subcommittee III-A to revise the specifications.

The subcommittee is currently revising the definitions of several terms and defining additional terms as revisions of the present glossary (B 243 – 55). Studies are in progress toward improved definitions of "cermets," "green strength" (as applied to metal powder specifications), "green density" and "*K*-factor".

Subcommittee II on Metal Powders (J. J. Cordiano, chairman).—Section A on Base Metal Powders (J. J. Cordiano, chairman) prepared the Tentative Method of Test for Green Strength of Compacted Metal Powder Specimens referred to earlier in this report. Efforts to establish a suitable method for determining compressibility of metal powders are continuing with improved precision and reproducibility as objectives.

The subcommittee approved the recommendation of the Advisory Committee of E-3 that a subcommittee be established in Committee E-3 (Methods of Chemical Analysis of Metal) with full representation from the producers and fabricators of metal powders to develop methods of chemical analysis of metal powders. Since Committee E-3 did not consider the method for determining the loss of weight in hydrogen heat treatment of metal powders, a chemical analysis, the subcommittee is preparing the method for letter ballot.

This committee approved the continuance of the Method of Subsieve Analysis of Granular Metal Powders by Air Classification B 293 – 54 T as tentative to allow additional time for the general use of the method.

Section B on Refractory Metal Powders (W. H. Bleecker, chairman), is preparing a method for measuring metal powder particle size with the Fisher Subsieve Sizer. Though results of a round-robin test using the method on samples of tungsten powder were generally satisfactory, there were some objections raised. Further revisions will be made before balloting in the subcommittee.

The subcommittee will continue work on the turbidimetric method for particle size.

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

The whole problem of particle size determination in the 1- to 10-micron range was forwarded to the ASTM Administrative Committee on Research for inclusion in the bulletin "Some Unsolved Problems."

Subcommittee III on Metal Powder Products (R. P. Koehring, chairman).—Section A on Bearings (M. H. Meighan, chairman) is preparing a proposed size list for flanged sleeve-type bearings. Work is continuing on the "*K*-factor" and its possible correlation with microstructure. A task group headed by H. R. Biehl will carry on a study of machinability of sintered bronze bearings with the objective of correlating machinability, "*K*-factor," and microstructure of sintered bronze parts.

Section B on Structural Parts (P. J. Shipe, chairman) prepared the Specifications for High-Density Iron, Sintered Metal Powder Structural Parts and the Specifications for Low- and Medium-Density Iron, Sintered Metal Powder Structural Parts referred to earlier in this report for publication as tentatives. A task group is improving the information contained in the appendix of each specification. Under consideration are changes in the 10-day rejection clause contained in the specifications prepared by the subcommittee and deleting the clause that states, "hardness tests are unreliable." Mechanical properties of brass as reported in the appendix of Specifications B 282 - 53 T is under investigation. The subcommittee voted to continue the two Specifications B 255 - 54 T and B 282 - 53 T as tentatives pending proposed changes.

Section C on Cemented Carbides (E. W. Engle, chairman) prepared and submitted for publication by the Society the Method of Test for Density of Cemented Carbides referred to earlier in this report. The subcommittee also recommended retaining the Recommended Practice for Hardness Testing of Cemented Carbides (B 294 - 54 T) as tentative pending investigation of errors that have been reported. The task groups on Carbide Classifications and Microstructure have been discharged. R. Dorr of the Carboloy Dept., General Electric Co., was appointed chairman of the task group on Physical Properties replacing J. G. Gurland, who has resigned from Subcommittee III-C.

Section E on Machinable Heavy Metals (A. S. Doty, chairman), newly organized, held two meetings during the year and is preparing a specification for machinable high-density tungsten-nickel-copper alloys. The scope of this specification was established and a task group prepared the first draft of a specification, which will be circulated to the section prior to the next meeting. It was decided that additional members for Section E would be solicited, particularly from consumers such as the aircraft industry.

This report has been submitted to a letter ballot of the committee, which consists of 71 members: 52 members returned their ballots, of whom 47 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. L. BONANNO,
Chairman.

C. G. JOHNSON,
Secretary.

REPORT OF ADVISORY COMMITTEE
ON
CORROSION*

The Advisory Committee on Corrosion held two meetings during the year: on November 1, 1955, at ASTM Headquarters, and on March 1, 1956, during Committee Week in Buffalo, N. Y.

PERSONNEL

D. A. George has been appointed as the alternate representative from Committee D-14 on Adhesives; and H. F. Hormann has been appointed as the alternate representative from Committee A-5 on Corrosion of Iron and Steel, replacing J. G. Thompson, recently retired. Committee B-4 on Metallic Materials for Electrical Heating, Electrical Resistance, and Electrical Contacts has appointed G. F. Geiger in addition to Chairman E. I. Shobert, II.

FINANCIAL STATUS

Although it will be necessary to increase the number of specimen racks and pipe frames at various locations to provide for programs currently under way or authorized, this can be done and still stay well within the original plans. In 1949 it was estimated that a fund of $100,000 would suffice for a ten-year period. After seven years of operation there is still more than $60,000 available. It is planned that in order to augment the present balance and in view of the recent use of facilities by Committee C-19 on Structural Sandwich Constructions, which group was not previously approached, an appeal be made to those who would benefit from the C-19 program.

TEST SITE CHANGES

New York, N. Y., Area:

The establishment of a heliport adjacent to the test site area located on the sixteenth floor penthouse roof of the Port of New York Authority Building in New York has been considered sufficient reason for the transfer of certain specimens to a new site on grounds owned by the U. S. Steel Supply Division, U. S. Steel Corp., in Newark, N. J. The site is near the Kearny test site of the U. S. Steel Corp. which was previously calibrated against the Port Authority location. Little difference was found with respect to degree of corrosivity. Arrangements have been made for fencing this area and for the construction and erection of six pipe frame supports.

It has been decided to refer to the Port Authority Building, Bell Laboratory roof, and the new site in Newark as "New York Area."

Mohave Desert:

A year ago Committee B-6 on Die-Cast Metals and Alloys had tentative plans for using an area provided by the Naval Installation at Inyokern (China Lake), Calif. This was not intended as an official ASTM test site and as the B-6 plans have not crystallized as anticipated, there will probably be no use made of this area by the Society.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

Key West, Fla.:

Establishment of a housing development in the immediate vicinity of the Key West test site resulted either in demolition or vandalism of virtually all the specimens remaining at that location. There will appear in the July ASTM BULLETIN an article by K. G. Compton indicating why, when considering past tests, it would be more proper to refer to the Key West environment as "Rural (Marine)" rather than "Marine."

EXPOSURE PROGRAMS IN PLANNING STAGE

Committee A-5 on Corrosion of Iron and Steel:

Subcommittee XVI on Hardware Tests has completed its plans for a new exposure test of hardware involving atmospheric exposure tests in the New York Area and Kure Beach, N. C. These specimens will include zinc and aluminum coatings on carbon and alloy steels.

Committee A-7 on Malleable Iron Castings:

This committee is planning a rather extensive program consisting of more than a thousand specimens to include:

1. Standard malleable.
2. Standard malleable, galvanized.
3. Standard malleable, black with 0.25 to 0.50 per cent copper added.
4. Low combined carbon, pearlitic malleable iron.
5. Relatively high combined carbon, pearlitic malleable iron.

Present plans call for exposure in the New York Area; Kure Beach, N. C.; East Chicago, Ind.; Brazos River, Tex.; and Point Reyes, Calif.

Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys:

It appears that this program which has been in the planning stage for some time will reach the exposure stage this fall. Specimens will include 3 by 6-in. and 4 by 6-in. panels, wires, spot-welded specimens, arc-welded specimens, and specimens of corrosion-resistant tubing. Sites to be used are Kure Beach, New York Area, State College, and Brazos River.

Committee B-8 on Electrodeposited Metallic Coatings:

A number of cadmium-plated steel panels for service testing of supplementary chromate films will be exposed at Kure Beach, Bell Telephone Laboratories (New York City), and at the Rock Island Arsenal in Rock Island, Ill. A number of the test panels will be provided with buttons of dissimilar metals for study of galvanic action. These metals will include brass, copper, and stainless steel.

Federation of Paint and Varnish Production Clubs:

The Federation of Paint and Varnish Production Clubs had been granted permission to use the Brazos River test site; this application was subsequently changed to Kure Beach, and this request has been approved pending availability of facilities.

CORROSION EXHIBIT

The Advisory Committee on Corrosion sponsored at the 1952 Annual Meeting a display representative of much of the Society's work in corrosion nad deterioration. In the past four years the number of programs and committees concerned have greatly increased and it is planned to hold a similar exhibit in

Atlantic City in June and at the Pacific Area National Meeting in Los Angeles in September.

Contributions from Committee A-5 on Corrosion of Iron and Steel will include a series of 9 by 12-in. Kodachrome prints of the sheet test specimens from the hardware tests, and a selected group of Kodachrome slides pertaining to the A-5 work.

Committee A-10 on Iron-Chromium, Iron-Chromiun-Nickel and Related Alloys will exhibit specimens of 17 per cent chromium and 18 per cent chromium-8 per cent nickel steels taken from the New York site. The Committee A-10 contribution will be supplemented by specimens from long-time tests on corrosion-resistant steels as conducted by several of the leading stainless steel producers.

Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys will make available the data from the work of Subcommittee VII on Weather, particularly the subgroup on test site calibration.

Committee B-7 on Light Metals and Alloys is contributing a number of specimens which have been tested after several years of exposure, and a series of blown-up charts summarizing data to date.

Committee B-8 on Electrodeposited Metallic Coatings has a display which will show the relative corrosivity of various environments on copper-nickel-chromium and on lead platings.

Committee C-19 on Structural Sandwich Constructions will have specimens of materials similar to those under test at State College and Kure Beach.

Committee D-1 on Paint, Varnish, Lacquer, and Related Products will have a number of metal panels showing the effects of one and two coat paint systems of varying formulae.

There will be a special section devoted to the effects of corrosion in high-purity water. This has been developed through the efforts of Committee D-19 on Industrial Water.

Respectfully submitted on behalf of the committee,

JEROME STRAUSS,
Chairman.

J. S. PETTIBONE,
Secretary.

REPORT OF COMMITTEE C-1

ON

CEMENT*

Committee C-1 on Cement held two meetings during the year: in Atlantic City, N. J., on June 30, 1955, and in Montreal, Quebec, Canada, on October 4, 1955. Special features of the Montreal meetings were planned tours of local industry and a technical session devoted to the development of the St. Lawrence Seaway and related topics.

The committee records with sorrow the death of three members: Harmon S. Meissner, Grant J. Durant, and F. O. Anderegg. Mr. Meissner, member since 1946, represented the Bureau of Reclamation; Mr. Durant, member since 1936, represented Froehling & Robertson, Inc., of Richmond, Va.; Mr. Anderegg, consulting engineer, was a member since 1940.

In recognition of their long association with Committee C-1 and their outstanding contributions to its work, the following two individuals were made Honorary Members of the committee: Perry J. Freeman, member for over 30 years; and John R. Dwyer, member since 1920 and Technical Assistant to Committee C-1 since 1931. During the year, the eleven Honorary Members were presented with certificates and citations.

At the 1955 Annual Meeting of the Society two members of Committee C-1 were honored by the Society: W. C. Hanna, Vice-Chairman of C-1, as a 50-year member, and H. F. Clemmer as a 40-year member.

The officers elected for the ensuing term of two years are as follows:

Chairman, R. R. Litehiser.
Vice-Chairman, W. C. Hanna.
Secretary, W. S. Weaver.

During the year, John R. Dise was appointed to succeed J. R. Dwyer as Technical Assistant to Committee C-1 and Supervisor of the Cement Reference Laboratory.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1955 Annual Meeting, Committee C-1 presented to the Society through the Administrative Committee on Standards the recommendation that the Standard Specifications for Masonry Cement (C 91 – 55) be revised and reverted to tentative status.

This recommendation was accepted by the Standards Committee on September 15, 1955, and the revised specifications appear in the 1955 Book of ASTM Standards, Part 3.

REVISION OF TENTATIVES

The committee recommends that the following Specifications and Methods be revised as indicated and continued as tentative:

Tentative Specifications for Portland Blast-Furnace Slag Cement (C 205 – 53 T):[1]

Table II.—Change the compressive strength requirements to read as follows for the various ages:

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] 1955 Book of ASTM Standards, Part 3.

Age, days	Types IS	IS-A
3	1200	900
7	2100	1500
28	3500	2800

Tentative Method of Test for Calcium Sulfate in Hydrated Portland Cement Mortar (C 265 – 55 T):[1]

Section 1.—Change the last sentence to read as follows:

> This method of test is intended for use primarily by manufacturers of portland cement and those interested in research on a suitable method for determining whether calcium sulfate has or has not been used in an amount considered to be optimum.

Section 8.—Delete the note at the end of this section.

Tentative Method of Test for False Set of Portland Cement (C 359 – 55 T):[1]

Section 2 (b).—Change to read as follows:

> Modified Vicat apparatus, as described in Section 2(*d*) of the Method of Test for Time of Setting of Hydraulic Cement by Vicat Needle (ASTM Designation: C 191), except that the 1-mm needle shall be replaced by a weight such that the total weight of the 1-cm plunger, indicator, and added weight shall be 400 ± 0.5 g.

Section 4 (d).—In line 8, change "2¾ min" to read "3 min."

Methods of Chemical Analysis of Portland Cement (C 114 – 51 T):[1]

Delete Sections 6 through 11 covering Darex Air-Entraining Agent.

This recommendation is made because an investigation conducted during the past few years has indicated that a suitable revision would require special complex apparatus and analytical precautions that do not seem warranted based on the minor use reported for the test.

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the following tentative be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Method of Test for Compressive Strength of Hydraulic Cement Mortars (Using 2-In. Cube Specimens) (**C 109 – 54 T**).[1]

ADOPTION OF TENTATIVE REVISION AS STANDARD

The committee recommends that the tentative revision relating to Section 7 of the Standard Method of Test for Autoclave Expansion of Portland Cement (**C 151 – 54**)[1] be approved for reference to letter ballot of the Society for adoption as standard.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following four methods of test be continued as tentative without revision. All are subjects of current study within the committee.

Tentative Method of Test for:

Potential Alkali Reactivity of Cement-Aggregate Combinations (**C 227 – 52 T**),
Time of Setting of Hydraulic Cement by Gillmore Needles (**C 266 – 51 T**),
Flexural Strength of Hydraulic Cement Mortars (**C 348 – 54 T**), and
Compressive Strength of Hydraulic Cement Mortars (Using Portions of Prisms Broken in Flexure) (**C 349 – 54 T**).

WITHDRAWAL OF STANDARD

The committee recommends that the Standard Method of Test for Soundness of Hydraulic Cement Over Boiling Water (Pat Test) (**C 189 – 49**)[1] be withdrawn since it is no longer used in any of the ASTM specifications.

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions of the following standards as indicated, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that these revisions may be referred to letter ballot of the Society. These revisions will bring Specifications C 150 and C 175 into more complete agreement with the soon-to-be-established Federal specifications for the corresponding cements.

Standard Specifications for Portland Cement (C 150 – 55):

Table II—Physical Requirements.—Change the values of the fineness requirements to read as follows:

	Type I	Type II	Type III	Type IV	Type V
Fineness, specific surface, sq cm per g (alternate methods):					
Turbidimeter test:					
Average value, min	1600	1600	...	1600	1600
Minimum value, any one sample included in the average	1500	1500	...	1500	1500
Air permeability test:					
Average value, min	2800	2800	...	2800	2800
Minimum value, any one sample included in the average	2600	2600	...	2600	2600

Standard Specifications for Air-Entraining Portland Cement (C 175 – 55):[1]

Table II—Physical Requirements.—Change the values of the fineness requirements to read as follows:

	Type IA	Type IIA	Type IIIA
Fineness, specific surface, sq cm per g (alternate methods):			
Turbidimeter test:			
Average value, min	1600	1600	
Minimum value, any one sample included in the average	1500	1500	
Air permeability test:			
Average value, min	2800	2800	
Minimum value, any one sample included in the average	2600	2600	

Change the strength requirements for Type IIIA cement to read as follows:

Compressive strength, psi (not less than):	
1 day in moist air	1300
1 day in moist air, 2 days in water	2500

EDITORIAL CHANGES

The committee recommends editorial changes in the following standards as indicated:

Standard Method of Test for Fineness of Portland Cement by Air Permeability Apparatus (C 204 – 55):[1]

Section 3 (e).—Replace the last four words of the second sentence with the statement "rod slightly smaller than the cell diameter."

Tentative Specifications for Portland-Blast Furnace Slag Cement (C 205 – 53 T):[1]

Table I.—Delete the requirements for TDA and 109-B. This action is recommended because it was not the intent of this specification that these constituents be determined to check compliance of a cement with specifications C 205.

Table II.—In the second sentence of Footnote *a*, change "Equation 10" to read "Equation 7 or 8." This change is necessary because of a renumbering of the equations in Method C 204.

Section 3 (*b*).—Delete the words "within the respective limits shown in Table I." This deletion is necessary because of the above revision in Table I.

Tentative Method of Test for Bleeding of Cement Pastes and Mortars (C 243 - 55 T):[1]

Section 4 (*b*).—Change to read as follows:

> (*b*) Mixing.—Mixing shall be done in conformance with Tentative Method for Mechanical Mixing of Hydraulic Cement Mortars of Plastic Consistency (ASTM Designation: C 305) except that:
>
> 1. Mixer shall be operated at low speed throughout.
> 2. There shall be a waiting period of 3 min between preliminary and final mixing in order to eliminate the effect of thixotropic set.
> 3. Both preliminary and final mixing shall be for a period of 3 min each.
>
> When mixing pastes, the materials for the batch shall be introduced in the following manner:
>
> 1. Place all the mixing water in the bowl.
> 2. Add the cement to the water and mix for 3 min at low speed,
> 3. Let the paste stand for 3 min. During the first 15 sec of this interval, quickly scrape down into the batch any paste that may have collected on the side of the bowl; then for the remainder of this period, cover with a damp cloth or lid.
> 4. Mix for 3 min at low speed.

Section 5.—Replace the last sentence with the following: "Of the final batch giving the proper flow, discard that portion used in the flow determination. Do not use it in the bleeding determination."

Section 7 (*b*).—Change the first sentence to read: "Immediately following the flow test, remix for 30 sec the mortar remaining in the mixing bowl."

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will reported at the Annual Meeting.[2]

This report has been submitted to letter ballot of the committee, which consists of 91 voting members; 79 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

R. R. LITEHISER,
Chairman.

W. S. WEAVER,
Secretary.

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[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-1 presented to the Society through the Administrative Committee on Standards the recommendation that the Tentative Specifications for Masonry Cement (C 91 - 55 T) be revised. This recommendation was accepted by the Standards Committee on December 17, 1956, and the revised tentative specifications appear in the 1956 Supplement to Book of ASTM Standards, Part 3.

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions of the following standards as indicated, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that these revisions may be referred to letter ballot of the Society. These revisions will bring Specifications C 150 and C 175 into more complete agreement with the soon-to-be-established Federal specifications for the corresponding cements.

Standard Specifications for Portland Cement (C 150 – 55):

Table II—Physical Requirements.—Change the values of the fineness requirements to read as follows:

	Type I	Type II	Type III	Type IV	Type V
Fineness, specific surface, sq cm per g (alternate methods):					
Turbidimeter test:					
Average value, min	1600	1600	...	1600	1600
Minimum value, any one sample included in the average	1500	1500	...	1500	1500
Air permeability test:					
Average value, min	2800	2800	...	2800	2800
Minimum value, any one sample included in the average	2600	2600	...	2600	2600

Standard Specifications for Air-Entraining Portland Cement (C 175 – 55):[1]

Table II—Physical Requirements.—Change the values of the fineness requirements to read as follows:

	Type IA	Type IIA	Type IIIA
Fineness, specific surface, sq cm per g (alternate methods):			
Turbidimeter test:			
Average value, min	1600	1600	
Minimum value, any one sample included in the average	1500	1500	
Air permeability test:			
Average value, min	2800	2800	
Minimum value, any one sample included in the average	2600	2600	

Change the strength requirements for Type IIIA cement to read as follows:

Compressive strength, psi (not less than):	
1 day in moist air	1300
1 day in moist air, 2 days in water	2500

EDITORIAL CHANGES

The committee recommends editorial changes in the following standards as indicated:

Standard Method of Test for Fineness of Portland Cement by Air Permeability Apparatus (C 204 – 55):[1]

Section 3 (e).—Replace the last four words of the second sentence with the statement "rod slightly smaller than the cell diameter."

Tentative Specifications for Portland-Blast Furnace Slag Cement (C 205 – 53 T):[1]

Table I.—Delete the requirements for TDA and 109-B. This action is recommended because it was not the intent of this specification that these constituents be determined to check compliance of a cement with specifications C 205.

Table II.—In the second sentence of Footnote *a*, change "Equation 10" to read "Equation 7 or 8." This change is necessary because of a renumbering of the equations in Method C 204.

Section 3 (*b*).—Delete the words "within the respective limits shown in Table I." This deletion is necessary because of the above revision in Table I.

Tentative Method of Test for Bleeding of Cement Pastes and Mortars (C 243 - 55 T):[1]

Section 4 (*b*).—Change to read as follows:

(*b*) Mixing.—Mixing shall be done in conformance with Tentative Method for Mechanical Mixing of Hydraulic Cement Mortars of Plastic Consistency (ASTM Designation: C 305) except that:

1. Mixer shall be operated at low speed throughout.
2. There shall be a waiting period of 3 min between preliminary and final mixing in order to eliminate the effect of thixotropic set.
3. Both preliminary and final mixing shall be for a period of 3 min each.

When mixing pastes, the materials for the batch shall be introduced in the following manner:

1. Place all the mixing water in the bowl.
2. Add the cement to the water and mix for 3 min at low speed,
3. Let the paste stand for 3 min. During the first 15 sec of this interval, quickly scrape down into the batch any paste that may have collected on the side of the bowl; then for the remainder of this period, cover with a damp cloth or lid.
4. Mix for 3 min at low speed.

Section 5.—Replace the last sentence with the following: "Of the final batch giving the proper flow, discard that portion used in the flow determination. Do not use it in the bleeding determination."

Section 7 (*b*).—Change the first sentence to read: "Immediately following the flow test, remix for 30 sec the mortar remaining in the mixing bowl."

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will reported at the Annual Meeting.[2]

This report has been submitted to letter ballot of the committee, which consists of 91 voting members; 79 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

R. R. LITEHISER,
Chairman.

W. S. WEAVER,
Secretary.

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[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-1 presented to the Society through the Administrative Committee on Standards the recommendation that the Tentative Specifications for Masonry Cement (C 91 - 55 T) be revised. This recommendation was accepted by the Standards Committee on December 17, 1956, and the revised tentative specifications appear in the 1956 Supplement to Book of ASTM Standards, Part 3.

REPORT OF COMMITTEE C-2
ON
MAGNESIUM OXYCHLORIDE AND MAGNESIUM OXYSULFATE CEMENTS*

Committee C-2 on Magnesium Oxychloride and Magnesium Oxysulfate Cements met once during the year: on June 28, 1955, in Atlantic City, N. J., during the Annual Meeting of the Society.

The committee consists of 17 members, of whom 7 are classified as producers, 3 as consumers, and 7 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, E. S. Newman.
Vice-Chairman, J. B. James.
Secretary, K. M. Berg.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1955 Annual Meeting, Committee C-2 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revisions of Tentative Specifications for:

Oxychloride Magnesia (C 275 – 51 T), and

Tentative Definitions of Terms for:

Magnesium Oxychloride and Magnesium Oxysulfate Cements (C 376 – 55 T).

These recommendations were accepted by the Standards Committee on February 6, 1956, and on September 22, 1955, respectively.

This report has been submitted to letter ballot of the committee, which consists of 19 members; 16 members have returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

D. S. HUBBELL,
Chairman.

K. M. BERG,
Secretary.

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*Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-2 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Method of Test for:

Yield of Magnesium Oxychloride Cement (Field Test) (C 388 – 56 T), and
Yield of Magnesium Oxychloride Cement (Laboratory Test) (C 389 – 56 T).

These recommendations were accepted by the Standards Committee on November 15, 1956, and the new tentative methods appear in the 1956 Supplement to Book of ASTM Standards, Part 3.

REPORT OF COMMITTEE C-3

ON

CHEMICAL-RESISTANT MORTARS*

Committee C-3 on Chemical-Resistant Mortars held two meetings during the year: on June 29, 1955, in Atlantic City, N. J., and on October 31, 1955, in Philadelphia, Pa.

The membership of the committee consists of 32 members, of whom 12 are classified as producers, 8 as consumers, 10 as general interest, and two as consulting members.

The officers elected for the ensuing term of two years are as follows:

Chairman, Beaumont Thomas.
Vice-Chairman, J. R. Allen.
Secretary, E. A. Reineck.

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Method of Test for Bond Strength of Chemical Resistant Mortars (**C 321 – 54 T**)[1] be approved for reference to letter ballot of the Society for adoption as standard without revision.

This recommendation has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

SUBCOMMITTEE ACTIVITIES

During the year the committee was reorganized; four "S" subcommittees were set up to write specifications on hydraulic, resin, silicate, and sulfur mortars, and four "T" subcommittees were set up to write test methods on mechanical, physical, and chemical properties, and recommended practices.

Subcommittee T-1 on Mechanical Properties (J. R. Allen, chairman) has developed a test method for measuring the compressive strength of silicate mortars which are now under consideration.

Subcommittee T-4 on Recommended Practices (W. H. Burton, chairman) has drafted recommendations for installing sulfur mortar joints, now under consideration.

This report has been submitted to letter ballot of the committee, which consists of 27 members; 27 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

BEAUMONT THOMAS,
Chairman.

E. A. REINECK,
Secretary.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] 1955 Book of ASTM Standards, Part 3.

[2] The letter ballot vote on this recommendation was favorable; the results of the vote are on record at ASTM Headquarters.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-3 presented to the Society through the Administrative Committee on Standards the recommendation that the proposed Recommended Practice for Installing Sulfur Mortar Joints be accepted for publication as tentative. This recommendation was accepted by the Standards Committee on September 5, 1956, and the new tentative recommended practice appears in the 1956 Supplement to Book of ASTM Standards, Part 3, bearing the designation C 386 – 56 T.

REPORT OF COMMITTEE C-4
ON
CLAY PIPE*

Committee C-4 on Clay Pipe held one meeting during the year: in Atlantic City, N. J. on June 28, 1955, at the time of the Annual Meeting of the Society.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1955 Annual Meeting, Committee C-4 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specifications for:
Extra Strength Clay Pipe (C 200 - 53 T), and
Extra Strength Ceramic Glazed Clay Pipe (C 278 - 51 T).
Tentative Revisions of Standard Specifications for:
Standard Strength Clay Sewer Pipe (C 13 - 54),
Standard Strength Perforated Clay Pipe (C 211 - 50), and
Standard Strength Ceramic Glazed Clay Sewer Pipe (C 261 - 54).

These recommendations were accepted by the Standards Committee on September 15, 1955, and the new and revised specifications appear in the 1955 Book of ASTM Standards, Part 3.

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

Committee C-4 recommends that the Tentative Specification for Clay Flue Linings (**C 315 - 53 T**)[1] be approved for reference to letter ballot of the Society for adoption as standard without revision.

AMERICAN STANDARDS

Since the last report (1954) of Committee C-4, the following standards under its jurisdiction were approved as American Standard by the American Standards Association:

Specifications for:
Standard Strength Perforated Clay Pipe (C 211 - 50),
Standard Strength Clay Pipe (C 13 - 54), and
Standard Strength Ceramic Glazed Clay Pipe (C 261 - 54).
Methods of Testing:
Clay Pipe (C 301 - 54).
Recommended Practice for:
Installing Clay Pipe (C 12 - 54).

This report has been submitted to letter ballot of the committee, which consists of 29 members; 26 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. C. RIEDEL,
Chairman.

R. G. SCOTT,
Secretary.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17-22, 1956.

[1] 1955 Book of ASTM Standards, Part 3.

REPORT OF COMMITTEE C-7

ON

LIME*

Committee C-7 on Lime held two meetings during the year: in Atlantic City, N. J., on July 1, 1955, at the time of the Annual Meeting of the Society, and in Buffalo, N. Y., on February 28, 1956, during ASTM Committee Week.

The committee consists of 54 members, of whom 22 are classified as producers, 10 as consumers, and 20 as general interest members, with 2 honorary members.

NEW TENTATIVE

The committee recommends that the Specifications for Fly Ash for Use as a Pozzolanic Material With Lime be accepted for publication as tentative as appended hereto.[1]

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions of the Standard Methods of Sampling, Inspection, Packing, and Marking of Quicklime and Lime Products (**C 50 - 27**)[2] as appended hereto, and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society.[3]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

SUBCOMMITTEE ACTIVITIES

Subcommittee II on Structural Lime (B. L. Corson, chairman) plans to review the Specifications for Quicklime for Structural Purposes (C 5 - 26).

Subcommittee III on Lime for the Chemical Industries (R. W. McAllister, chairman) is developing specifications for lime for use in the manufacture of silica brick, sand-lime brick, and hypochlorite bleach.

Subcommittee V on Methods of Test of Lime and Lime Products (D. G. Hamme, chairman) is working on two tests for lime neutralization.

Subcommittee VII on Pozzolans (L. J. Minnick, chairman) prepared the Specifications for Fly Ash for Use as a Pozzolanic Material With Lime, referred to earlier in the report. The subcommittee is planning to develop additional detailed specifications for various uses of lime and pozzolanic material.

Subcommittee IX on Research (W. C. Voss, chairman) presented a six-month progress report of round robin tests on the expansive characteristics of masons' lime by autoclave testing procedure.

This report has been submitted to letter ballot of the committee, which consists of 54 members; 48 members returned their ballots, of whom 31 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

JAMES A. MURRAY,
Chairman.

ROBERT S. BOYNTON,
Secretary.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] The new tentative appears in the 1956 Supplement to Book of ASTM Standards, Part 3.

[2] 1955 Book of ASTM Standards, Part 3.

[3] This recommendation was not accepted at the annual meeting, see Summary of Proceeding, p 28.

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquaters.

REPORT OF COMMITTEE C-8

ON

REFRACTORIES*

Committee C-8 on Refractories held two meetings during the year: the eighty-eighth at Bedford Springs, Pa., on October 6, 1955; and the eighty-ninth at Buffalo, N. Y., on March 2, 1956.

The committee lost by death Stephen M. Swain and Earl C. Petrie both of the North American Refractories Co.

The committee consists of 44 members, of whom 23 are classified as producers, 14 as consumers, and 7 as general interest members. The membership limit has been increased from 45 to 46.

The officers elected for the ensuing term of two years are as follows:

Chairman, J. J. Hazel.
Vice-Chairman, S. M. Phelps.
Secretary, L. J. Trostel.

NEW TENTATIVE

The committee recommends for publication as tentative the Methods for Chemical Analysis of Refractory Materials, as appended hereto.[2] These methods are in effect a tentative revision of, and, when adopted, are intended to be added to the Standard Methods for Chemical Analysis of Refractory Materials (C18 – 52).

TENTATIVE REVISIONS OF STANDARDS

The committee recommends tentative revisions of the following standards as indicated:

Standard Methods of Chemical Analysis of Refractory Materials (C 18 – 52):[1]

Sections 16, 27, and 39.—Add the following Note to the end of each of these sections:

NOTE.—There is an alternate method for K_2O and Na_2O, which includes Li_2O as well; this method will be found in Section 20 of the Tentative Methods for Chemical Analysis of Refractory Materials (ASTM Designation: C18 – 56T).[2]

Standard Definitions of Terms Relating to Refractories (C 71 – 55):[1]

Delete the tentative revision issued in 1955 covering the definition for nine inch series brick.

Add the following 10 new definitions:

Raw Refractory Dolomite.—Granular natural dolomite that is sufficiently low in fluxing constituents for use as a refractory material.

Calcined Refractory Dolomite.—Granular refractory dolomite that has been heated only enough to convert the calcium and magnesium carbonates into oxides.

Dead Burned Refractory Dolomite.—Granular refractory dolomite that has been burned (fired) above 1540 C with or without additives so as to form primarily lime and periclase in a matrix that provides resistance of the grains to hydration and carbonation.

Abutment.—The structural portion of a furnace which withstands the thrust of an arch.

Acid Proof Brick.—Brick having low porosity and permeability, and a high resistance to chemical attack by most acids.

Air Set.—The property of a material to develop high strength upon loss of moisture.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] 1955 Book of ASTM Standards, Part 3.

[2] The new tentative appears in the 1956 Supplement to Book of ASTM Standards, Part 3.

Air Ramming.—A method of forming refractory shapes, furnace hearths, or other furnace parts by means of pneumatic hammers.

American Bond.—Brick work in which stretcher courses are the principal portion, with headers laid every fifth, sixth, or seventh course.

Arch.—In furnace construction, a structure which usually spans two walls, and may be supported by them.

Arch Brick.—A brick having the largest faces inclined at the same angle toward one narrow side.

ADOPTION OF TENTATIVE REVISIONS AS STANDARD

The committee recommends that tentative revisions issued June, 1955, of the Standard Method of Test for Pyrometric Cone Equivalent (P.C.E.) of Refractory Materials (**C 24 – 46**)[1] be approved for reference to letter ballot of the Society for adoption as standard.

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions of the following standards as indicated, and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Method of Test for Disintegration of Fireclay Refractories in an Atmosphere of Carbon Monoxide (C 288 – 54):[1]

Title.—Delete the word "fireclay."

Section 1.—In line 3, delete the word "fireclay."

Section 3.—Add the following Paragraph (*c*):

> (*c*) Unfired refractories may be prepared for testing by firing above 1000 F and under suitable conditions to remove combined water, and organic and volatile matter.

Section 4(*a*).—In the first sentence, change "without the carbon monoxide atmosphere" to read "in a nitrogen atmosphere."

Note 1.—In line 3, change the word "air" to read "nitrogen". Change the last sentence to read: "A second method consists in flushing the nitrogen from the system with a fast flow of carbon monoxide."

The purpose of the foregoing revisions is to make the method applicable to unfired refractories and to carbon and carbon-ceramic refractories.

Standard Classification of Fireclay Refractories (C 27 – 41):[1]

The proposed revised classification was published as information only in the 1955 Proceedings, p. 397. At the October, 1955, meeting of the committee it was recommended that the revised classification be published as a tentative revision of C 27 – 41, which action was confirmed by letter ballot. Further action was taken by the committee at its meeting in March, 1956, recommending that the revision be adopted as standard. The revised classification has not been published by the Society in the form of the tentative revision since this recommendation was received subsequent to the preparation of Part 3 of the 1955 Book of Standards, and therefore the revised classification is offered for immediate adoption.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

EDITORIAL REVISIONS

The committee recommends editorial revisions of the following standards as indicated:

Standard Method of Test for Pyrometric Cone Equivalent (P.C.E.) of Refractory Materials (C 24 – 46):[1]

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[3] The letter ballot on these recommendations was favorable; the results of the vote are on record at ASTM Headquaters.

Fig. 3.—Replace with a new illustration of the plaque showing the new cone numbers, and particularly a corrected representation of the 82-deg angle of inclination.

Appendix.—In the tentative revision of the Appendix, change the third paragraph to read: "Standard pyrometric cones (P.C.E.) of numbers 28 and 30 are manufactured but are not used in the P.C.E. test."

Standard Specification for Refractories for Malleable Iron Furnaces with Removable Bungs, and for Annealing Ovens (C 63 – 51):[1]

Section 8 (c).—Change "cone no. 31–32" to read "cone no. 31½."

Section 8 (d).—Change "an intermediate" to read "a medium."

Standard Specifications for Refractories for Heavy Duty Stationary Boiler Service (C 64 – 51):[1]

Sections 6 (a), (b), (c), (e).—Change "cone no. 31–32" to read "cone no. 31½."

Section 6 (f).—Change "an intermediate" to read "a medium."

Standard Specifications for Refractories for Incinerators (C 106 – 51):[1]

Section 5 (a).—Change "cone no. 31–32" to read "cone no. 31½."

Section 5 (b).—Change "an intermediate" to read "a medium."

Specifications for Refractories for Moderate Duty Stationary Boiler Service (C 153 – 51):[1]

Section 5 (a).—Change "an intermediate" to read "a medium."

Tentative Method of Test for Permanent Linear Change on Firing of Castable Refractories (C 269 – 55 T):[1]

Section 1.—Delete the word "hard" before "fireclay base."

Tentative Classification of Single- and Double-Screened Ground Refractory Materials (C 316 – 53 T):[1]

Section 5 (a).—In the second paragraph, change the first sentence to read: "If the shipment consists of 100 containers or less, two shall be selected at random and their contents dumped and riffled to provide a 5-pound sample from each." Change line 14 of the second paragraph to read: "The two or more 5-pound samples . . ."

Section 6.—In line 11, delete the words "at least." Change the last sentence to read: "The per cent figures from the respective sieves shall check within 2 per cent in duplicate determinations; otherwise these determinations shall be repeated."

Tentative Method of Test for Bulk Density of Granular Refractory Materials (C 357 – 55 T):[1]

Section 1.—Add to the end of this section: "This method is not suitable for materials which, in accordance with paragraph 6 (*b*), may hydrate in boiling water."

Section 5 (f).—Delete the words "such as dead-burned magnesite."

Section 6 (b).—In line 1, following the word "ignition," add: "(according to 5 (*f*))."

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Industrial Survey (R. P. Stevens, chairman) has supplied one revised and two new surveys for the 1956 Manual, and will revise the surveys that are more than ten years old.

Subcommittee III on Tests (S. M. Phelps, chairman): Section A on Load (A. J. Metzger, chairman) is continuing its modulus-of-rupture studies on bricks of other forms than the standard orthorhombic; studies are also continuing on the hot load test using smaller specimens than the present standard. The section is still looking for a laboratory to study the problem of temperature gradients in the hot load test.

Section B on Spalling (L. J. Trostel, chairman) finds a kaolin-alumina mix better than kaolin alone as a mortar for the high-temperature panel. Improvements in the pyrometry of the spalling panel, and a test method for spalling of blast-furnace brick are under consideration.

Section C on Temperature (J. L. Carruthers, chairman) has been requested to investigate further the effects of furnace and fuel, leading possibly to the specification of a single type of furnace for the P.C.E. test.

Section E on Chemical Analysis (H. A. Heiligman, chairman) has completed a 3-year study of the application of flame photometry to the determination of alkalies in refractories, and has cooperated with Subcommittee XII in preparing procedures for the analysis of carbon and carbon-ceramic refractories. The section is continuing work on a hydration test for basic refractories. The section will supply the material for two proposed new standard samples of silica brick to be issued by the National Bureau of Standards.

Section F on Refractory Insulation (W. L. Stafford, chairman) is collecting data for a specification for insulating fire brick used in furnaces in which atmospheric reactions are negligible. These data will be turned over later to Subcommittee VII.

Section G on Porosity and Permanent Volume Change (L. C. Hewitt, chairman) will look into the feasibility of preparing a standard test for permeability of refractories to gases, and the adoption of a unit therefor.

Section H on Mortars and Plastic and Castable Refractories (G. H. Anthony, chairman) is giving further attention to a method for consistency to replace the rather crude procedure now contained in Methods C 268 and C 269.

Section J on Carbon Monoxide Disintegration (H. M. Kraner, chairman) in cooperation with Subcommittee XII, has modified the CO test to cover carbon and carbon-ceramic refractories.

Subcommittee VI on Nomenclature (W. S. Debenham, chairman) finds no consensus of usage for the term "nine-inch series brick" and the tentative definition has been withdrawn. The glossary prepared by The Refractories Institute is being reviewed for revision and addition to Standard Definitions C 71.

Subcommittee VII on Specifications (J. D. Sullivan, chairman) has under consideration modifications of Specification C 213, which may be offered for adoption when a proposed new classification of castable refractories is accepted.

Subcommittee VIII, Editorial (R. B. Sosman, chairman) is preparing an eighth edition of the Manual for publication in 1956.

Subcommittee IX on Classifications (A. H. Couch, chairman) is at work on a classification of silica brick. A proposed classification of castable refractories prepared by The Technical Advisory Committee of The Refractories Institute is recommended for publication as information only.[3]

Section A on Pouring Pit Refractories for Steel (J. A. Roslund, chairman) has been reassigned the problem of classifying ladle brick. Some members of the committee have expressed the belief that ladle brick are unclassifiable, indicating that the significant properties of a ladle brick are completely unknown, a conclusion that the committee is not yet ready to accept.

Subcommittee XI on Special Refractories (H. C. Fisher, chairman) cooperating with the Special Refractories Association and its Standards Subcommittee I, has prepared a Classification of Mullite Refractories which is now being considered by the manufacturers of mullite brick.

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[3] See p. 207.

Subcommittee XII on Carbon Refractories (E. B. Snyder, chairman) finds a lack of interest in a test for rate of oxidation. However, a reheat test is being prepared. Data are being assembled on a test for permeability to gases.

Subcommittee XV on Basic Granular Refractories (J. J. Hazel, chairman) prepared definitions and a classification for granular dolomitic refractories. The definitions as revised by Subcommittee VI are set forth previously in the report, and the classification is under consideration by Subcommittee IX. Chemical and physical test methods are being assembled.

Special Subcommittee on Lime for Silica Brick (L. J. Trostel, chairman) has proposed chemical and physical tests for the Standard Specification for Quicklime and Hydrated Lime for Silica Brick Manufacture (C 49 - 42), which is under jurisdiction of Committee C-7 on Lime.

This report has been submitted to letter ballot of the committee, which consists of 44 members; 44 members returned their ballot, of whom 43 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

R. B. SOSMAN,
Chairman.

W. R. KERR,
Secretary.

APPENDIX

SUGGESTED CLASSIFICATION OF CASTABLE REFRACTORIES[1]

SUBMITTED BY C-8 SUBCOMMITTEE IX[2]

Scope

1. This classification pertains to castable refractories that, when tempered with water, will develop structural strength by reason of a hydraulic set.

NOTE.—This classification does not apply to special castable refractories such as the high-strength types or gun mixes.

Classes of Alumina-Silica-Base Castable Refractories

2. The regular alumina-silica-base castable refractories are classified on the basis of volume stability of cast test brick when fired at the temperatures specified in Table I.

Classes of Insulating Castable Refractories

3. Insulating castable refractories are classified on the basis of bulk density of dried cast test brick and volume stability when fired as specified in Table II.

Methods of Testing

4. The properties enumerated in this classification shall be determined in accordance with the following methods of test:

(*a*) *Permanent Linear Shrinkage.*—Method of Test for Permanent Linear Change on Firing of Castable Refractories (ASTM Designation: C 269).[3]

(*b*) *Bulk Density* (*Weight per Cubic Foot*).—Methods of Test for Size and Bulk Density of Refractory Brick (ASTM Designation: C 134)[3] on test brick prepared in accordance with the Method of Test for Permanent Linear Change on Firing of Castable Refractories (ASTM Designation: C 269)[3] and after the 220 to 230 F oven-drying of Section 7 of Method C 269.

TABLE I.—REGULAR CASTABLE REFRACTORIES.

Test Requirements	Classes of Alumina-Silica-Base Castables				
	Class 21	Class 24	Class 26	Class 28	Class 30
Permanent linear shrinkage not more than 1.5 per cent when fired for 5 hr at........	2000 F	2300 F	2500 F	2700 F	2900 F

TABLE II.—INSULATING CASTABLE REFRACTORIES.

Test Requirements	Classes of Insulating Castable Refractories			
	Class 18	Class 20	Class 22	Class 24
Permanent linear shrinkage not more than 1.5 per cent when fired for 5 hr at....................	1700 F	1900 F	2100 F	2300 F
Maximum bulk density (lb per cu ft) after drying at 220 to 230 F..........................	55	65	75	90

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[1] This suggested classification has no official status in the Society, but is published as information only. Comments are solicited.

[2] Submitted by Subcommittee IX on Classifications, of the ASTM Committee C-8 on Refractories.

[3] 1955 Book of ASTM Standards, Part 3.

REPORT OF COMMITTEE C-9

ON

CONCRETE AND CONCRETE AGGREGATES*

Committee C-9 on Concrete and Concrete Aggregates held two meetings during the year: on June 28, 1955, during the Annual Meeting of the Society in Atlantic City, N. J., and on October 7, 1955, in Montreal, Quebec, Canada.

During the year the committee adopted a memorial to Mr. Grant J. Durant, a member of the committee since 1939 who died on October 22, 1955.

The Sanford E. Thompson Award, established by the committee in 1938 in honor of its first chairman, is made for a paper of outstanding merit on concrete or concrete aggregates presented before the Society. As a result of revision in the procedures for consideration of papers for ASTM awards which requires that the papers published during any one year will be reviewed during the year following publication for award consideration, the papers which, under the previous procedures, would have been considered for an award this year will be considered for a possible award to be made at the 1957 Annual Meeting and accordingly no award will be made this year.

REVISION OF TENTATIVES

The committee recommends that the following tentatives be revised as indicated and continued as tentative:

Tentative Specifications for Concrete Aggregates (C 33 – 55 T):[1]

Section 4(c).—Change to read as follows:

(*c*) Fine aggregate for use in concrete which will be subject to frequent wetting, extended exposure to humid atmosphere, or contact with moist ground shall not contain materials which are deleteriously reactive with the alkalies in the cement in an amount sufficient to cause excessive expansion of mortar or concrete except that if such materials are present in injurious amounts, the fine aggregate may be used with a cement containing less than 0.6 per cent alkalies calculated as sodium oxide or with the addition of a material that has been shown to prevent harmful expansion due to alkali-aggregate reaction.

NOTE.—A number of methods for detecting potential reactivity have been proposed. However, they do not provide quantitative information on the degree of reactivity to be expected or tolerated in service. Therefore, evaluation of potential reactivity of an aggregate should be based upon judgment and on the interpretation of test data and examination of concrete structures containing a combination of fine and coarse aggregates and cements for use in the new work. Results of the following tests will assist in making the evaluation:

(*1*) *Recommended Practice for Petrographic Examination of Aggregates for Concrete* (*ASTM Designation: C 295*).—Certain materials are known to be reactive with the alkalies in cements. These include the following forms of silica: opal, chalcedony, tridymite and cristobalite; intermediate to acid (silica-rich) volcanic glass such as is likely to occur in rhyolite, andesite, or dacite; certain zeolites such as heulandite; and certain constituents of some phyllites. Determination of the presence and quantities of these materials by petrographic examination is helpful in evaluating potential alkali reactivity. Some of these materials render an aggregate deleteriously reactive when present in quantities as little as 1.0 per cent or even less.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 18–22, 1956.

[1] 1955 Book of ASTM Standards, Part 3.

(*2*) *Tentative Method of Test for Potential Reactivity of Aggregates (Chemical Method) (ASTM Designation: C 289)*.—In this test, where R_c signifies reduction in alkalinity and S_c dissolved silica, both in millimoles per liter, the following criteria may be used to evaluate potential reactivity:

(*a*) If R_c exceeds 70, the aggregate is considered potentially reactive if S_c is greater than R_c.

(*b*) If R_c is less than 70, the aggregate is considered potentially reactive if S_c is greater than $35 + \frac{R_c}{2}$.

The test can be made quickly and, while not completely reliable in all cases, provides helpful information, especially where results of the more time-consuming tests are not available.

(*3*) *Tentative Method of Test for Potential Alkali Reactivity of Cement-Aggregate Combinations (ASTM Designation: C 227)*.—The results of this test, when made with a high alkali cement furnish information on the likelihood of harmful reactions occurring. The alkali content of the cement should be substantially above 0.6 per cent and preferably above 0.8 per cent, expressed as sodium oxide. Combinations of aggregate and cement which have produced excessive expansions in this test usually should be considered potentially reactive. While the line of demarcation between non-reactive and reactive combinations is not clearly defined, expansion is generally considered to be excessive if it exceeds 0.05 per cent at 3 months or 0.10 per cent at 6 months. Expansions greater than 0.05 per cent at 3 months should not be considered excessive where the 6-month expansion remains below 0.10 per cent. Data for the 3-month tests should be considered only when 6-month results are not available.

(*4*) *Tentative Method of Test for Potential Volume Change of Cement-Aggregate Combinations (ASTM Designation: C 342)*.—Cement-aggregate combinations tested by this procedure whose expansion equals or exceeds 0.200 per cent at an age of one year may be considered unsatisfactory for use in concrete exposed to wide variations of temperature and degree of saturation with water.

Section 8(b).—Change to read as indicated for Section 4(*c*) except that the word "fine" should be changed to read "coarse" and instead of the note indicated to follow Section 4(*c*), insert the following:

NOTE.—Methods for evaluating the reactivity of aggregates are discussed in the note to Section 4(*c*).

Tentative Method of Test for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (C 88 – 55 T):[1]

Section 7(d).—Add the following note:

NOTE.—A piece of aggregate is defined as any fragment which weighs at least 10 per cent of the oven dry weight of the fragment from which it broke.

Tentative Specifications for Lightweight Aggregates for Insulating Concrete (C 332 – 54 T):[1]

New sections.—Add the following new Sections 6 and 7 and renumber the subsequent sections accordingly:

Packaging and Marking

6. When lightweight aggregates covered by this specification are delivered in packages, the name of the manufacturer, type of aggregate, and minimum weight and approximate volume of the contents shall be plainly indicated thereon.

Rejection

7. (*a*) The purchaser of materials covered by this specification shall have the option of evaluating these materials for rejection, by either minimum weight or approximate volume as stated.

(*b*) Individual packages may be rejected when:

(*1*) The contents, on a weight basis, is 5 per cent less than that indicated on the package, or

(*2*) The contents, on a volume basis, is 10 per cent less than that indicated on the package.

(*c*) The entire shipment may be rejected:

(*1*) On a weight basis when the average contents of two packages for each 100 but not less than 6 packages selected at random, in any one shipment, is less than that indicated on the package.

(*2*) On a volume basis when the average contents of two packages for each 100 but not less than 6 packages selected at random, in any one shipment differs by more than 5 per cent from that indicated on the package.

(*d*) The net weight of the contents shall be determined by weighing the package or packages and deducting the weight of the container.

(*e*) The volume of the contents in the package shall be calculated by determining the weight of the contents of the package and then obtaining the weight per cu ft of the aggregate; from an average weight package of the samples selected in Section 7(*c*) (*2*); by the procedure given in Standard Method of Test for Unit Weight of Aggregate (ASTM Designation C 29) (Shovelling Method) Sections 7(*a*) and 7(*b*) and then dividing the weight of the contents of the bag by the weight per cu ft of aggregate.

Tentative Method of Test for Resistance of Concrete Specimens to Rapid Freezing and Thawing in Water (C 290 - 52 T):[1]

Section 2(a). — In paragraph (*2*), change the word "Note" to read "Note 1" and add the following Note 2:

NOTE 2.—Each specimen should be supported at the bottom of its container in such a way that the temperature of the heat-exchanging medium will not be transmitted directly through the bottom of the container to the full area of the bottom of the specimen, thereby subjecting it to conditions substantially different from the remainder of the specimen. A flat spiral of $\frac{1}{8}$-in. wire, or a paraffin-treated wood block $\frac{1}{2}$ to $\frac{5}{8}$ in. thick placed in the bottom of the container has been found adequate for this purpose.

Section 2.—Delete Paragraph (*c*) and reletter the subsequent paragraphs accordingly.

Section 4.—Add the following Note:

NOTE.—This test method contemplates the use of specimens not less than 3 in. nor more than 5 in. in width and depth and not less than 14 in. nor more than 16 in. in length. Difficulty may be experienced in complying with the requirements of the test method if specimens are used which differ appreciably from the above dimensions.

Add the following to the end of this section:

For this test the specimens shall be stored in saturated lime water from the time of their removal from the molds until the time freezing and thawing tests are started. All specimens to be compared with each other shall be of the same initial dimensions.

Section 5(b).—Add as the fourth sentence: "The specimens shall be returned to random positions in the apparatus."

Add to the end of this section: "Each time a specimen is tested for fundamental frequency a note shall be made of its visual appearance and special comment made on any defects which develop."

Section 7(a).—Add the following to the end of this section:

(8) Any defects in each specimen present at 0 cycles of freezing and thawing.

Section 7(c).—Add the following to the end of this section:

Any defects in each specimen which are present before the start of the test or which develop during testing, and the number of cycles at which such defects were noted.

Tentative Method of Test for Resistance of Concrete Specimens to Rapid Freezing in Air and Thawing in Water (C 291 - 52 T):[1]

Section 2(a). — In Paragraph (*2*), change the word "Note" to read "Note 1" and add the following Note 2:

NOTE 2.—The supports should be such that they are not in contact with the full area of the supported side or end of the specimen, thereby subjecting this area to conditions substantially different from the remainder of the specimen. The use of relatively open gratings, metal rods, or the edges of metal angles has been found adequate for this purpose provided the heat-exchanging medium can circulate in the direction of the long axis of the rods or angles.

Section 2.—Delete Paragraph (*c*) and reletter the subsequent paragraphs accordingly.

Section 3.—Add a new Paragraph (*c*) to read as follows and reletter the subsequent paragraphs accordingly:

(*c*) The difference between the temperature at the center of a specimen and the temperature at its surface shall at no time exceed 50 F.

In the present Paragraph (*d*), change the first sentence to read: "Conformity with requirements of Paragraphs (*a*), (*b*), and (*c*) shall be based on . . . ."

Section 4.—Change as recommended above for Method C 290.

Section 5(*b*). — Change as recommended above for Method C 290.

Section 7(*a*). — Change as recommended above for Method C 290.

Section 7(*c*). — Change as recommended above for Method C 290.

Tentative Method of Test for Resistance of Concrete Specimens to Slow Freezing and Thawing in Water or Brine (C 292 - 52 T):[1]

Section 2(*a*). — Change as recommended above for Method C 290.

Section 2.—Delete Paragraph (*c*) and reletter the subsequent paragraphs accordingly.

Section 4.—Change as recommended above for Method C 290.

Section 5(*b*). — Change as recommended above for Method C 290.

Section 7(*a*). — Change as recommended above for Method C 290.

Section 7(*d*). — Change as recommended for Section 7(*c*) of Method C 290.

Tentative Method of Test for Resistance of Concrete Specimens to Slow Freezing in Air and Thawing in Water (C 310 - 53 T):[1]

Section 2(*a*).—Add the following Note to the end of Paragraph (*2*):

NOTE.—When specimens are tested without containers, the supports should be such that they are not in contact with the full area of the supported side or end of the specimen, thereby subjecting this area to conditions substantially different from the remainder of the specimen. Similarly when specimens are tested in containers they should be supported in such a way that the temperature of the heat-exchanging medium will not be transmitted directly through the bottom of the metal container to the bottom of the specimen. The use of relatively open gratings, metal rods, or the edges of metal angles have been found adequate for supporting specimens without containers provided the heat-exchanging medium can circulate in the direction of the long axis of the rods or angles. A flat spiral of $\frac{1}{8}$-in. wire or a paraffin-treated wood block $\frac{1}{2}$ to $\frac{5}{8}$ in. thick placed in the bottom of the container has been found adequate for supporting specimens in containers.

Section 2.—Delete Paragraph (*c*) and reletter the subsequent paragraphs accordingly.

Section 3.—Change as recommended above for Method C 291.

Section 4.—Change as recommended above for Method C 290.

Section 5(*b*). — Change as recommended above for Method C 290.

Section 7(*d*). — Change as recommended above for Section 7(*c*) of Method C 290.

Tentative Method of Test for Bleeding of Concrete (C 232 - 53 T):[1]

Section 2(*a*).—Add the following to the end of this section:

The container shall be made of metal having a thickness of No. 10 to No. 12 U. S. gage and shall be reinforced around the top with a No. 10 to No. 12 U. S. gage metal band 1$\frac{1}{2}$ in. wide. The inside shall be smooth and free from corrosion, coatings, or lubricants.

Section 2(*b*).—Change to read as follows:

(*b*) A scale of sufficient capacity to weigh the load required with an accuracy of 0.5 per cent.

Section 3(*a*).—Change to read as follows:

(*a*) For concrete made in the laboratory prepare the concrete as described in Sections 2, 3, and 4 of the Standard Method of Making and Curing Concrete Compression and Flexure Specimens in the Laboratory (ASTM Designation C 192).

For concrete made in the field, sample the concrete as described in Standard Method of Sampling Fresh Concrete (ASTM Designation C 172).

Revision of Standard and Reversion to Tentative

The committee recommends that the following standards be revised as indicated and reverted to tentative status:

Standard Method of Test for Compressive Strength of Molded Concrete Cylinders (C 39 - 49):[1]

Section 2.—Replace the fourth, fifth, and sixth sentences with the following:

The bearing faces shall not depart from a plane by more than 0.001 in. in any 6 in. for blocks 6 in. in diameter or larger or by more than 0.001 in. in the diameter of any smaller block; and new blocks shall be manufactured within one-half of this tolerance. The maximum diameter of the bearing face of the suspended spherically seated block shall not exceed the values given below:

Diameter of Test Specimen, in.	Maximum Diameter of Bearing Face, in.
2	4
3	5
4	6½
6	10
8	11

The center of the sphere shall coincide with the center of the bearing face. If the radius of the sphere is smaller than the radius of the bearing face, the portion of the bearing face extending beyond the sphere shall have a thickness not less than the difference in radii. The diameter of the bearing face shall be at least as great as the diameter of the sphere. The bearing faces shall be at least as large and preferably slightly larger than the surface of the specimen to which the load is applied. When the diameter of the bearing face exceeds the diameter of the specimen by ½ in. or more, concentric circles shall be inscribed to facilitate proper centering.

Standard Method of Test for Organic Impurities in Sands for Concrete (C 40 - 48):[1]

Revise as appended hereto.[2]

Standard Method of Test for Air Content of Freshly Mixed Concrete by the Pressure Method (C 231 - 54):[1]

Section 1.—Change the first two sentences to read as follows:

1. This method of test covers the determination of the air content of freshly mixed concrete from observations of the change in volume with a change in pressure. A detailed procedure employing one type of apparatus is described. Apparatus of other designs has been developed and appropriate procedures have been prepared for use with such apparatus.

Section 2.—Change to read as follows:

2 (*a*). All types of apparatus employed in determining the air content of freshly mixed concrete by the pressure method must, in order to provide satisfactory results, incorporate the following:

(*1*) A measuring bowl of sufficiently rigid construction to make a pressure-tight container of accurate volume and suitable to hold a representative sample of concrete to be tested.

(*2*) A cover designed to be attached to the measuring bowl so as to make an adequately rigid, pressure tight assembly.

(*3*) Facilities for applying a known pressure to the system and observing its effect on the volume of the sample.

(*4*) Appropriate tools for placing and consolidating the sample and using the apparatus.

(*b*) One satisfactory set of apparatus is described below:

(*1*) *Measuring Bowl.*—The measuring bowl shall be a flanged cylindrical bowl, preferably of steel or other hard metal not readily attacked by the cement paste, having a diameter equal to 0.75 to 1.25 times the height. The outer rim and upper surface of the flange, as well as the interior surfaces of the bowl, shall be smooth machined surfaces. The capacity of the measuring bowl shall be at least as large as is specified in the following table (Note 1), depending on the size of the aggregate in the concrete:

Minimum Capacity of Measuring Bowl, cu ft	Nominal Maximum Size of Aggregate, in.
0.20	2
0.40	3
2.50	6

The measuring bowl and cover assembly shall be pressure tight and sufficiently rigid to limit the expansion factor, *D*, of the apparatus assembly (Section 4(*d*)) to not more than 0.1 per cent of air content on the standpipe indicator scale when under the normal operating pressure.

Note 1.—Larger containers should be used for larger samples of concrete in order to reduce errors of sampling.

(*2*) *Conical Cover Assembly.*—The cover shall be flanged, preferably composed of steel or other hard metal not readily attacked by the cement paste and shall have interior surfaces inclined not less than 30 degrees from the surface of the flange. The outer rim and lower surface of the flange and the sloping interior shall be such that the cover and the measuring bowl can be fitted together into a pressure tight assembly. The cover shall be sufficiently rigid to limit the expansion factor of the apparatus assembly as prescribed in Paragraph (*b*) (*1*).

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[2] The new tentative appears in the 1956 Supplement to Book of ASTM Standards, Part 3.

The cover shall be fitted with a standpipe which may be a graduated precision-bore glass tube or may be metal of uniform bore with a glass water gage attached. The graduations for a suitable range in air content shall be in per cent and tenths of a per cent as determined by the proper air pressure calibration test. The internal diameter of the standpipe shall be selected so that under the normal operating pressure the water column will be lowered sufficiently to measure air contents to 0.1 per cent (Note 2). The applied air pressure shall be indicated by a pressure gage connected to the air chamber above the water column. The gage shall have suitable graduations and a full scale reading twice the normal working pressure (Note 3).

The cover shall be fitted with a suitable device for venting of the air chamber, an air valve, and a petcock for bleeding off water as required. Suitable means for clamping the cover to the bowl shall be provided to make a pressure-tight seal without entrapping air at the joint between the flanges of the cover and bowl. A suitable hand pump shall be provided with the cover, either as an attachment or as an accessory.

Note 2.—It is suggested that approximately a 1-in. lowering of the water column should represent 1 per cent of air content.

Note 3.—Pressures of 7.5 to 30 psi have been used satisfactorily. However, each container shall be calibrated for a stated normal working pressure.

(*3*) *Calibration Cylinder.*—The calibration cylinder shall consist of a cylindrical measure having an internal volume equal to approximately 3 to 6 per cent of the volume of the measuring bowl. A satisfactory measure may be machined from No. 16 gage brass tubing (of proper diameter to provide the volume desired) to which a brass disk ¼ in. in thickness is soldered to form an end.

(*4*) *A Coil Spring* or other means shall be provided for holding the calibration cylinder in place.

(*5*) *A Tube* of appropriate diameter shall be provided either as an integral part of the cover assembly or separately so constructed that when water is added to the container, there will be a minimum of disturbance to the concrete.

(*6*) *A Trowel* of the standard brick mason's type shall be provided.

(*7*) *Tamping Rod.*—A round, straight, steel rod ⅝ in. in diameter and approximately 24 in. in length having one end rounded to a hemispherical tip whose diameter is ⅝ in.

(*8*) *A Mallet* with a rubber or rawhide head, weighing ½ lb for containers smaller than 0.50 cu ft capacity and 1 lb or more for larger containers, shall be provided.

(*9*) *A Strike-Off Bar* consisting of a flat straight steel bar shall be provided.

(*10*) *A Funnel* with spout fitting into the tube described in Paragraph (5) shall be provided.

(*11*) *A Measure* having a ½- or 1-gal capacity, as required, shall be provided to fill the indicator with water from the top of the concrete to the zero mark.

Section 3.—Add a new Section 3 on Procedure as follows and renumber the present Section 3 and subsequent sections:

3. The procedures set forth in Sections 4, 5, 6, and 7 for calibration of the apparatus, determination of aggregate correction factor, determination of air content of concrete, and calculation of results apply specifically to the use of the apparatus described in Section 2(*b*). When apparatus of alternative design is employed, procedures appropriate to its use shall be followed. The intent of the procedures outlined below will serve as a guide to development of similar procedures for use with other types of equipment.

Present Section 3.—First sentence—Delete "under job conditions."

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives which have stood without revision for two or more years, be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Descriptive Nomenclature of Constituents of Natural Mineral Aggregates (**C 294 – 54 T**),[1] and

Tentative Specifications for Liquid Membrane-Forming Compounds for Curing Concrete (**C 309 – 53 T**).[1]

ADOPTION OF TENTATIVE REVISIONS AS STANDARD

The committee recommends that the tentative revisions of the following standards be approved for reference to letter ballot of the Society for adoption as standard:

Standard Definitions of Terms Relating to Concrete and Concrete Aggregates (**C 125 – 48**),[1] (Tentative Revisions issued June, 1954).
Standard Specifications for Waterproof Paper for Curing Concrete (**C 171 – 53**).[1]

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following tentatives, which have stood without revision for two or more years be continued as tentative:

Tentative Methods of Test for:

Potential Reactivity of Aggregates (Chemical Method) (**C 289 – 54 T**),
Volume Change of Cement Mortar and Concrete (**C 157 – 54 T**),
Volume Change of Concrete Products (**C 341 – 54 T**),
Lightweight Pieces in Aggregate (**C 123 – 53 T**),

Tentative Methods of:

Sampling and Testing Fly Ash for Use as an Admixture in Portland-Cement Concrete (**C 311 – 54 T**),

Tentative Specifications for:

Fly Ash for Use as an Admixture in Portland-Cement Concrete (**C 350 – 54 T**),
Lightweight Aggregates for Structural Concrete (**C 330 – 53 T**), and
Lightweight Aggregates for Concrete Masonry Units (**C 331 – 53 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

This report has been submitted to letter ballot of the committee, which consists of 127 active members commanding 98 votes, plus 6 honorary members having voting privileges; 124 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. H. PRICE,
Chairman.

BRYANT MATHER,
Secretary.

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[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-9 presented to the Society through the Administrative Committee on Standards the recommendation that the proposed Specifications for Packaged, Dry, Combined Materials for Mortar and Concrete be accepted for publication as tentative. This recommendation was accepted by the Standards Committee on September 5, 1956, and the new tentative specifications appear in the 1956 Supplement to Book of ASTM Standards, Part 3, bearing the designation C 387 – 56 T.

REPORT OF COMMITTEE C-11

ON

GYPSUM*

Committee C-11 on Gypsum held two meetings during the year: in Atlantic City, N. J., on June 28, 1955, and in Buffalo, N. Y., on February 28 and 29, 1956.

The committee consists of 32 members, of whom 13 are classified as producers, 9 as consumers, and 10 as general interest members.

The officers and members-at-large on the Executive Committee elected for the ensuing term of two years are as follows:

Chairman, G. W. Josephson.
Vice-Chairman, B. W. Nies.
Secretary, O. H. Storey, Jr.
Executive Committee Members-at-Large: Theodore Irving Coe, Frank L. Marsh, Stanton Walker, and J. Stanley Young.

Recommendations Accepted by the Administrative Committee on Standards

Subsequent to the 1955 Annual Meeting, Committee C-11 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Specification for:

Precast Reinforced Gypsum Slabs (**C 377 – 56 T**).

Tentative Revision of Standard Methods of Testing:

Gypsum and Gypsum Products (**C 26 – 54**).

These recommendations were accepted by the Standards Committee on January 27, 1956, and Specification C 377 is available as a separate reprint.

New Tentative

The committee recommends that the Specifications for Annular Ringed Nails for Gypsum Wallboard be accepted for publication as tentative as appended hereto.[1]

Revisions of Standard, Immediate Adoption

The committee recommends for immediate adoption the following revisions of the Standard Methods of Testing Gypsum and Gypsum Products (**C 26 – 54**)[2] and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Section 18(c).—Change to read as follows:

> (*c*) Standard sand shall be used exclusively and shall consist of a natural silica sand from Ottawa, Ill., graded to pass a No. 20 (840-micron) sieve and to be retained on a No. 30 (590-micron) sieve. This sand shall be considered standard when not more than 15 g are retained on the No. 20 sieve and not more than 5 g pass the No. 30 sieve after 5 min of continuous sieving of a 100-g sample.

Section 19.—Change the center heading immediately preceding Section 19 to read "Normal Consistency of Gypsum Plaster."

Change the note immediately following the center heading to read:

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] The new tentative appears in the 1956 Supplement to Book of ASTM Standards, Part 3.

[2] 1955 Book of ASTM Standards, Part 3.

> NOTE.—Since accuracy in determining normal consistency is most important in the standardizing of physical methods for testing cementitious materials. it is essential that this test be performed with great care.

Redesignate Paragraph (*d*) as new Section 21, renumbering the subsequent sections accordingly, and deleting "consistometer." The reference letters *A*, *B*, and *C* to Fig. 1 should also be deleted from this revised section.

Section 20.—Change the subheading immediately preceding Section 20 to "Apparatus" by deleting "Procedure for Gypsum Plaster." In line 3 of Paragraph (*a*), change the word "Vaseline" to read "petroleum jelly."

Add the following as Paragraphs (*c*) and (*d*), these paragraphs being modifications of the present Sections 22 (*a*) and (*b*):

> (*c*) Calcined gypsum shall be considered of normal consistency when a penetration of 30 ± 2 mm is obtained when tested in accordance with Section 20, weight of rod and plunger for this determination to be 50 g. Normal consistency shall be expressed as the number of milliliters of water required to be added to 100 g of the gypsum.
>
> (*d*) All gypsum mixtures containing aggregates shall be considered of normal testing consistency when a penetration of 20 ± 3 mm is obtained when tested in accordance with Section 20, weight of rod and plunger for these determinations to be 150 g. Normal consistency shall be expressed as the number of milliliters of water required to be added to 100 g of the mixture.

At the end of Section 20 add:

> NOTE.—Gypsum neat plaster is tested for compressive strength (Section 28 (*a*)) at a normal consistency determined after the plaster is first mixed with standard sand in the ratio of 200 g of sand to 100 g of plaster.

Section 21.—Insert a center heading preceding Section 21 to read: "Normal Consistency of Gypsum Concrete."

Change the subheading immediately preceding Section 21 by deleting "for Gypsum Concrete" and redesignate as Section 22.

Add the following as Paragraph (*d*), this Paragraph being a modification of the present Section 22(*c*):

> (*d*) Gypsum concrete shall be considered of normal consistency when a patty diameter of 15 ± ½ in. is obtained when tested in accordance with Section 21. Normal consistency shall be expressed as the number of milliliters of water required to be added to 100 g of the gypsum concrete.

Delete the note at the end of Section 21.

Change the first sentence of Paragraph 21(*b*) to read: "Sift 2000 g of the sample into a known volume of water to which 0.5 g commercial retarder has previously been added."

Section 22.—Delete Section 22, "Normal Consistency," which is replaced by the above revisions.

Section 23.—Delete Section 23, "Water Carrying Capacity" in its entirety, and renumber the subsequent sections accordingly.

Section 25(*a*).—Delete "All Calcined Gypsum Products" from the heading and substitute "Gypsum Concrete and All Gypsum Plasters."

Change the first two sentences to read: "Mix 200 g of the sample (Note) to make a paste of normal consistency. For the quantity of water and directions for mixing, see Sections 19 to 22, except that no retarder shall be added."

Delete the sixth sentence: "If the sample is not retarded, test it every 2 min until nearly set, and then every 1 min." Change the next sentence to read: "Test the sample at such intervals as are necessary to determine whether it complies with the requirements for time of setting for the product tested."

Section 25(*b*).—Change the first two sentences to read as follows:

> (*b*) *Gypsum Neat Plaster.*—Test gypsum neat plaster for time of setting as mixed with 3 parts by weight of standard sand that has been washed and dried in accordance with the procedure de-

scribed in Section 18(*c*). Mix dry a 100-g sample of the gypsum neat plaster and 300 g of the standard sand and then add sufficient water to produce a stiff yet workable mortar. Stir for 1 min to an even, lump-free consistency.

Section 26.—Add the following note at the end of this section:

NOTE.—For compressive strength *of gypsum concrete only*, 2 by 4-in. cylinder molds may be used instead of cube molds if desired. Prepare the specimens as described in Section 27, except that the paste shall be struck off to a smooth surface flush with the tops of the molds immediately after the molds are filled. Test the specimens and report the results as described for cube specimens in Sections 28 and 29.

Section 27(a).—Change this paragraph to read:

(*a*) Mix sufficient sample at normal consistency to produce not less than 1000 ml of mixed mortar and cast into six 2-in. split cube molds. Neat gypsum plaster shall be premixed dry with two parts by weight of standard sand (Section 18(*c*)). For the quantity of water, see Sections 19 to 22 except that no retarder shall be added. Place the required amount of water in a clean 2-qt mixing bowl. For all gypsum plasters except gypsum concrete, add the required amount of dry plaster and allow to soak for 2 min. Mix vigorously for 1 min with a metal spoon or stiff-bladed spatula to produce a mortar of uniform consistency. For gypsum concrete, soak for 1 min and stir vigorously for 3 min.

Section 27(b).—Insert before the first sentence "(*b*) The molds shall be coated with a thin film of mineral oil and placed on an oiled glass or metal plate." Insert after the present second sentence, "Cut off the excess mortar or paste to a plane surface flush with the top of the mold after the mortar has set, using a broad knife or similar implement."

Section 28.—Add the following:

Position the cubes in the testing machine so that the load is applied on surfaces formed by faces of the molds, not on top and bottom. Apply the load continuously and without shock, at a constant rate within the range 15 to 40 psi per sec. During application of the first half of the maximum load a higher rate of loading is permitted.

New Sections.—Add the following new Sections 41 and 42, renumbering the present and subsequent sections accordingly:

WEIGHT AND THICKNESS OF PAPER SURFACING OF GYPSUM BOARDS

41. *Test Specimens.* Cut a specimen approximately 7 in. square from the field of each of three sample boards. Place specimens on edge in an oven maintained at 250 to 300 F for not less than 4 hr and as much longer as may be required for the gypsum core to be softened by calcination. Then separate the face and back papers from the core by careful slitting, sawing, or peeling. Avoid tearing, splitting, or gouging of the paper. Remove adhering core material by brushing with a medium stiff brush. If desired, removal of core material can be facilitated by washing in a stream of water while brushing. Continue cleaning until paper is essentially free from adhering gypsum. If specimens have been washed, next dry them to constant weight at 100 to 110 F. Whether washed or not washed, then accurately trim them to 6 in. square. Then bring the specimens to normal weight by exposing them overnight to conditions of 60 to 85 F, 25 to 60 per cent relative humidity.

42. *Procedure.*—(*a*) Weigh the three specimens of face paper together, to 0.1 g and multiply their combined total weight in grams by 2.94 to obtain the weight in lb per 1000 sq ft. Similarly determine the weight of the back paper.

(*b*) Using a self-reading micrometer gage (see Section 44), measure and record the thickness of each specimen at three locations at least 4 in. apart. Average all the readings on the face paper specimens to obtain the thickness of the face paper. Average all the readings on the back paper specimens to obtain the thickness of the back paper.

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends that the Tentative Specifications for Inorganic Aggregates for Use in Interior Plaster (C 35 – 54 T) be continued as tentative without revision.

The recommendations in this report have been submitted to letter ballot of

the committee, the results of which will be reported at the Annual Meeting.[3]

This report has been submitted to letter ballot of the committee, which consists of 32 members; 24 members returned their ballots, of whom 23 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

G. W. JOSEPHSON,
Chairman.

O. H. STOREY, JR.,
Secretary.

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE C-12

ON

MORTARS FOR UNIT MASONRY*

Committee C-12 on Mortars for Unit Masonry held two meetings during the year: on June 30, 1955, in Atlantic City, N. J., and on February 29, 1956, in Buffalo, N. Y.

The committee consists of 40 members, of whom 17 are classified as producers, 11 as consumers, and 12 as general interest members.

The committee records with regret the death of F. O. Anderegg, Chairman of Subcommittee II on Research.

The Officers elected for the ensuing two-year term are as follows:

Chairman, R. E. Copeland.

First Vice-Chairman, Harry C. Plummer.

Second Vice-Chairman, P. M. Woodworth.

Secretary, C. U. Pierson, Jr.

TENTATIVES CONTINUED WITHOUT REVISION

Pending formal action on changes now under consideration, the committee recommends that the following three tentatives be continued without revision:

Tentative Specifications for:

Aggregate for Masonry Mortar (**C 144 - 54 T**),
Mortar for Reinforced Brick Masonry (**C 161 - 44 T**), and
Mortar for Unit Masonry (**C 270 - 54 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[1]

SUBCOMMITTEE ACTIVITIES

Subcommittee III on Specifications and Methods of Test for Mortar (H. C. Plummer, chairman) is working actively on revisions of Specifications C 161 - 44 T and C 270 - 54 T.

Subcommittee IV on Specifications for Aggregates for Mortar (Stanton Walker, chairman) is working on the evaluation of sands on a performance basis. Pending the results of these studies, it has been recommended that Specifications C 144 - 54 T be continued as tentative.

Working Subcommittee on Efflorescence (P. L. Rogers, chairman).—A set of three cements has been sent to cooperating laboratories for study of the effect of proposed modifications in the wick test for efflorescence on the reproducibility of results between laboratories.

This report has been submitted to letter ballot of the committee, which consists of 41 members; 32 returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

R. E. COPELAND,
Chairman.

J. A. MURRAY,
Secretary.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17-22, 1956.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE C-13

ON

CONCRETE PIPE*

Committeee C-13 on Concrete Pipe held one meeting during the year: in Chicago, Ill. on November 7 and 8, 1955.

The present membership of the committee consists of 42 members, of whom 20 are classified as producers, 15 as consumers, and 7 as general interest members.

The committee was reorganized at its November meeting to provide for an Advisory Committee and for standing subcommittees on each of the specifications under its jurisdiction. The Advisory Committee consists of the three officers and three members-at-large elected by the membership. The standing subcommittees are as follows:

Subcommittee I on Non-Reinforced Sewer Pipe (Specification: C14) (Carl A. Bluedorn, chairman).

Subcommittee II on Reinforced Sewer and Culvert Pipe (Specifications: C75 and C76) (C. M. Adams, chairman).

Subcommittee III on Irrigation and Drain Pipe (Specifications: C4 and C118) (P. W. Manson, chairman).

Subcommittee IV on Low-Head Pressure Pipe (Specification: C361) Howard G. Curtis, chairman).

Subcommittee V on Low-Head Internal Pressure Sewer Pipe (Specification: C362) (J. A. Dunn, chairman).

The Absorption Test Subcommittee was retained with J. W. Johnson appointed as chairman.

The officers and members-at-large of the Advisory Committee elected for the ensuing term of two years are as follows:

Chairman, R. R. Litehiser.

Vice-Chairman, E. F. Bespalow.

Secretary, Howard F. Peckworth.

Advisory Committee: Members-At-Large, J. A. Dunn, Merrill Butler and James W. Johnson.

In January, 1956 the Board of Directors took action to transfer jurisdiction for concrete drain tile from Committee C-15 on Manufactured Masonry Units to Committee C-13, leaving with Committee C-15 jurisdiction for clay drain tile. The Board requested Committees C-13 and C-15 to recommend effective means of coordination.

ADOPTION OF TENTATIVE REVISIONS AS STANDARD

The committee recommends that the tentative revisions relating to Sections 3, 9, 18, and 19 of the Standard Specifications for Concrete Sewer Pipe (**C 14 – 55**)[1] be approved for reference to letter ballot of the Society for adoption as standard with the following modification of the revision of Section 3:

Add the following footnote, with the reference to the footnote appearing after the word "permeability tests:"

> Prior to purchase, the purchaser may specify the hydrostatic test prescribed in Specification C 14 – 55 in lieu of the permeability test.

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revision of the following standards and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that these revisions may be referred to letter ballot of the Society:

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17-22, 1956.

[1] 1955 Book of ASTM Standards, Part 3.

Standard Specifications for Concrete Sewer Pipe (C 14 - 55):[1]

Section 17(b).—Change to read as follows:

> (b) *Drying Specimens.*—Specimens shall be dried in a ventilated oven at a temperature of 105 to 115 C (221 to 239 F) until two successive weighings at intervals of not less than 2 hours show an increment of loss not greater than 0.1 per cent of the original weight of the specimen.

Section 29(i).—Change to read as follows:

> (i) The complete absence of distinct web-like markings from the external surface of pipe made by any process in which the forms are removed immediately after the concrete has been placed, which is indicative of a deficiency of water in the concrete mix, unless all specimens submitted for test which do not have such web-like markings shall have passed the physical tests herein required.

Standard Specifications for Reinforced Concrete Sewer Pipe (C 75 - 55):[1]

Section 28(b).—Change to read as recommended above for Section 17(*b*) of Standard Specification C14.

Section 31(b).—Change to read as follows:

> (b) Variations of the position of the reinforcement shall not exceed one-tenth of the wall thickness but in no case shall the allowable variation be less than ¼-in. nor more than ⅝-in. from the position provided in the design; but the cover on the reinforcement shall be not less than ¾-in. at any point.

Section 36(f).—Change to read as recommended above for Section 29(*i*) of Standard Specification C14.

Standard Specifications for Reinforced Concrete Culvert Pipe (C 76 - 55):[1]

Section 30 (b).—Change to read as recommended above for Section 17(*b*) of Standard Specification C14.

Section 33(b).—Change to read as recommended above for Section 31(*b*) of Standard Specifications C75.

Section 38(f).—Change to read as recommended above for Section 29(*i*) of Standard Specification C14.

Standard Specifications for Concrete Irrigation and Drainage Pipe (C 118 - 55):[1]

Section 15(b).—Change to read as recommended above for Section 17(*b*) of Standard Specification C14.

Section 19(c).—Change to read as recommended above for Section 29(*i*) of Standard Specification C14.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

SUBCOMMITTEE ACTIVITIES

A great number of recommendations were made to the committee at its meeting in November 1955. The majority of these were turned over to the five subcommittees on standard specifications or the Absorption Test Subcommittee for study and recommendation at the next meeting of the committee.

The Task Group on Specifications for Low-Head Concrete Pressure Pipe was discharged being replaced by Subcommittees IV on Low-Head Pressure Pipe and V on Low-Head Internal Pressure Sewer Pipe.

This report has been submitted to letter ballot of the committee which consists of 42 members; 29 members have returned their ballots, of whom 26 have voted affirmatively and 2 negatively.

Respectfully submitted on behalf of the committee,

R. R. LITEHISER,
Chairman.

HOWARD F. PECKWORTH,
Secretary.

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[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE C-14

ON

GLASS AND GLASS PRODUCTS*

Committee C-14 on Glass and Glass Products held two meetings during the year: on September 16, 1955, in Bedford Springs, Pa., and on April 25, 1956, in New York, N. Y.

During the year there were 7 resignations from the committee, and 9 new members were added. Mr. Paul A. Webster, Emhart Corp., was lost to the committee by death during the year. The committee, as of April 25, consisted of 47 voting members, of whom 15 were classified as producers, 17 as consumers, and 15 as general interest members. There were 20 nonvoting members, and 10 consultants.

The officers elected for the ensuing term of two years are as follows:

Chairman, L. G. Ghering.
Vice-Chairman, Harold Simpson.
Secretary, F. V. Tooley.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1955 Annual Meeting, Committee C-14 presented to the Society through the Administrative Committee on Standards a tentative revision of the Standard Methods of Chemical Analysis of Glass Sand (C 146 – 43). The recommendation, which was accepted by the Standards Committee, on January 27, 1956, consisted of the addition of photometric methods of analysis, and they will be published under the designation C 146 – 56 T.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following two tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Methods of:

Sampling Glass Containers **(C 224 – 53 T)**,[1] and

Tentative Methods of Testing Structural Non-Load-Bearing Cellular Glass Blocks **(C 240 – 54 T)**.[1]

ADOPTION OF TENTATIVE REVISIONS AS STANDARDS

The committee recommends that the tentative revisions of the Standard Definitions of Terms Relating to Glass and Glass Products (**C 162 – 52**)[1] be approved for reference to letter ballot of the Society for adoption as standard.

The tentative revisions consist of new definitions for "annealing range" and "strain point," and the revision of definitions for "annealing point" and "softening point." These tentative revisions were issued December 22, 1954.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following tentatives, which have stood without revision for two or more years, be retained as tentative:

Tentative Method of Test for:

Annealing Point and Strain Point of Glass **(C 336 – 54 T)**,

Average Linear Expansion of Glass **(C 337 – 54 T)**, and

Softening Point of Glass **(C 338 – 54 T)**.

*** Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.**

[1] 1955 Book of ASTM Standards, Part 3.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

SUBCOMMITTEE ACTIVITIES

Subcommittee I on Nomenclature and Definitions (H. H. Holscher, chairman) reviewed the tentative revisions of Definitions C 162 – 52 referred to earlier in the report for adoption as standard.

Subcommittee II on Chemical Analysis (F. W. Glaze, chairman) prepared the tentative revision of the Methods of Chemical Analysis of Glass Sand (C 146 – 43) which was accepted through the Administrative Committee on Standards as noted earlier in the report. Round-robin tests have been planned for the purpose of refining the routine procedures in the Standard Methods of Chemical Analysis of Soda-Lime Glass (C 169 – 53).

Subcommittee VI on Glass Construction Block and Tile (A. H. Baker, chairman) recommended the adoption as standard without change of the Tentative Methods of Sampling and Testing Structural Non-Load-Bearing Cellular Glass Blocks (C 240 – 54 T) as noted earlier in the report.

Subcommittee VII on Glass Containers (R. F. Scott, chairman) is rewriting the Standard Methods of Polariscopic Examination of Glass Containers (C 148 – 50) to include two alternate methods; one being the present method involving the use of glass disks and the other being a new method using polarimeter apparatus. Seven laboratories are cooperating in round-robin tests and their recommendation is expected at the fall meeting of the committee.

This report has been submitted to letter ballot of the committee, which consists of 47 voting members; 22 members returned their ballots, of whom 21 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

L. G. GHERING,
Chairman.

F. V. Tooley,
Secretary.

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[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE C-15

ON

MANUFACTURED MASONRY UNITS*

Committee C-15 held two meetings during the year: on July 1, 1955, in Atlantic City, N. J., in conjunction with the Annual Meeting of the Society, and on March 1, 1956, in Buffalo, N. Y., during ASTM Committee Week.

The committee consists of 74 members of whom 36 are classified as producers, 17 as consumers, and 21 as general interest members.

On January 16, 1956, the Board of Directors of the Society transferred jurisdiction for concrete drain tile from Committee C-15 to Committee C-13 on Concrete Pipe. Subcommittee X of Committee C-15 will now be responsible for only clay drain tile.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, J. W. Whittemore.
First Vice-Chairman, Theodore I. Coe.
Second Vice-Chairman, F. M. Woodworth.
Secretary, M. H. Allen.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1955 Annual Meeting, the Committee presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Revision of Standard:

Specification for Building Brick (C 62 - 50), Specification for Facing Brick (C 216 - 50), and Methods of Sampling and Testing Brick (C 67 - 50).

Revision of Tentative:

Specification for Ceramic Glazed Structural Clay Facing Tile, Facing Brick, and Solid Masonry Units (C 126 - 52 T).

These recommendations were accepted by the Standards Committee on September 15, 1955 and the tentative revisions and revised tentative appear in the 1955 Book of ASTM Standards, Part 3.

ADOPTION OF TENTATIVE REVISION AS STANDARD

The committee recommends that the tentative revision of Section 7 of Standard Methods of Sampling and Testing Concrete Masonry Units (**C 140 - 55**)[1] be approved for reference to letter ballot of the Society for adoption as standard.

This recommendation has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Clay Brick and Structural Clay Tile (J. A. Lee, chairman).—Tentative revisions of the Standard Specifications for Building Brick (C 62 - 50), Facing Brick (C 216 - 50), and of Standard Methods of Sampling and Testing Brick (C 67 - 50), accepted during the year by the Administrative Committee on Standards, were prepared by the subcommittee. The subcommittee is continuing its study of the use of sulfur for capping compression test specimens of clay brick and structural clay tile, with a view to recommending sulfur capping as an alternate method of capping in Standard Methods of Sampling and Testing Brick (C 67 - 50) and Standard Methods of Sampling and Testing Structural Clay Tile (C 112 - 52).

Subcommittee III on Concrete and

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] 1955 Book of ASTM Standards, Part 3.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Sand Lime Units (R. E. Copeland, chairman) has recommended that the tentative revision of Section 7, Capping Compressive Test Specimens, of Standard Methods of Sampling and Testing Concrete Masonry Units (C 140 – 55) be adopted as standard after having remained tentative for two years. A study committee was appointed to review the need for developing a method of testing concrete masonry units for frost resistance. The subcommittee is developing a standard method for determining moisture content of concrete masonry units by the relative humidity technique and expects to present definite recommendations in the near future. Another study committee has recommended the preparation of a new specification for a class of concrete masonry units having low shrinkage and moisture volume change properties. The subcommittee is currently studying possible methods for determining shrinkage so that shrinkage limits distinguishing low shrinkage from normal shrinkage can be established.

Subcommittee V on Waterproofing Materials for Unit Masonry Walls, (C. C. Connor, chairman) has been studying methods for determining the effectiveness and durability of moisture proof coatings. Terminology related to waterproofing materials is also being studied.

Subcommittee VI on Glazed Brick and Tile, (E. F. Wanner, chairman) prepared the revisions to Tentative Specification for Ceramic Glazed Structural Clay Facing Tile, Facing Brick and Solid Masonry Units (C 126 – 52 T), accepted during the year by the Administrative Committee on Standards.

Subcommittee IX on Chemical Resistant Units, (V. W. Weidman, chairman) is working on specifications for Industrial Floor Brick, which it expects to complete by the time of the 1957 Committee Week, and for Ceramic Tower Packings, which probably will not be ready for action until 1958.

Subcommittee X on Drain Tile (J. G. Sutton, chairman) is being reorganized in light of the action of the ASTM Board of Directors in removing concrete drain tile from its jurisdiction. The future work of this subcommittee will be limited to clay drain tile.

This report has been submitted to letter ballot of the committee which consists of 74 members; 57 members have returned their ballots, of whom 56 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. W. WHITTEMORE,
Chairman.

M. H. ALLEN,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-15 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Revision of Standard Specifications for:

Building Brick (Solid Masonry Units Made from Clay or Shale) (C 62 – 50),
Hollow Load-Bearing Concrete (C 90 – 52),
Hollow Non-Load-Bearing Concrete Masonry Units (C 129 – 52), and
Solid Load-Bearing Concrete Masonry Units (C 145 – 52).

Tentative Revision of Standard Methods of:

Sampling and Testing Brick (C 67 – 50).

These recommendations were accepted by the Standards Committee on September 5, 1956, and the tentative revisions appear in the 1956 Supplement to Book of ASTM Standards, Part 3.

REPORT OF COMMITTEE C-16

ON

THERMAL INSULATING MATERIALS*

Committee C-16 on Thermal Insulating Materials and its subcommittees held two meetings during the year: at State College, Pa., on October 24 to 26, 1955, and at Buffalo, N. Y., on February 29, March 1 and 2, 1956.

The committee consists of 91 voting members, 24 non-voting members, 3 honorary members, 1 consulting member, and 3 associate members. The voting membership is classified as follows: 49 producers, 20 consumers, and 22 general interest members.

A new Subcommittee B on Definitions was formed with K. M. Ritchie as chairman.

The officers elected for the ensuing term of two years were as follows:

W. L. Gantz, Chairman.
C. B. Bradley, Vice Chairman.
W. C. Lewis, Vice Chairman.
J. M. High, Secretary.
R. A. Funger, Assistant Secretary.
E. C. Shuman, Assistant Secretary, Membership.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1955 Annual Meeting, Committee C-16 presented to the Society through the Administrative Committee on Standards, the recommendation that the Specification for Mineral Wool Block or Board Insulation (For Low Temperatures) be accepted for publication as tentative. This recommendation was accepted by the Standards Committee on January 27, 1956, and the new Specification will appear in the 1956 Supplement to Book of ASTM Standards, Part 3, bearing the designation C 378 - 56 T.

NEW TENTATIVES

The committee recommends that the following specifications and methods be accepted for publication as tentative as appended hereto:[1]

Tentative Specifications for:

Cellular Glass Thermal Insulation for Pipes, and
Mineral Wool Felt Insulation (Industrial Type) for Elevated Temperatures.

Tentative Method of Test for:

Wet Adhesion of Thermal Insulating Cements to Metal.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Specifications for:

Cellular Asbestos Paper Thermal Insulation for Pipes (**C 298 - 52 T**),[2]
Laminated Asbestos Thermal Insulation for Pipes (**C 299 - 52 T**),[2]
Cellular Glass Insulating Block (**C 343 - 54 T**),[2]
Corkboard Thermal Insulation (**C 352 - 54 T**).[2]

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17-22, 1956.

[1] These new tentatives appear in the 1956 Supplement to Book of ASTM Standards, Part 3.

[2] 1955 Book of ASTM Standards, Part 3.

Tentative Method of Test for:

Density of Preformed Pipe-Covering-Type Thermal Insulation (**C 302 – 52 T**),[2]
Density of Preformed Block-Type Thermal Insulation (**C 303 – 52 T**),[2]
Adhesion of Dried Thermal Insulating Cements (**C 353 – 54 T**),[2] and
Compression Strength of Thermal Insulating Cements (**C 354 – 54 T**).[2]

ADOPTION OF TENTATIVE REVISION AS STANDARD

The committee recommends that the tentative revision of the Standard Definitions of Terms Relating to Thermal Insulating Materials (**C 168 – 51**)[2] comprising three new definitions of water vapor transmission be approved for reference to letter ballot of the Society for adoption as Standard.

WITHDRAWAL OF TENTATIVES

The committee recommends the withdrawal of the Tentative Method of Test for Vibration Resistance of Preformed Thermal Insulation (**C 304 – 52 T**)[2] on the basis that this test is not required.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following tentatives which have stood without revision for two or more years be continued as tentative since they are undergoing review in the subcommittee with a view to revising them before their adoption to standard:

Tentative Specifications for:

Mineral Wool Molded-Type Pipe Insulation (for Elevated Temperatures) (C 281 – 52 T),
Mineral Wool Molded-Type Pipe Insulation (For Low Temperatures) (C 300 – 52 T),
Diatomaceous Earth Block-Type Thermal Insulation (C 333 – 54 T),
Diatomaceous Earth Thermal Insulation for Pipes (C 334 – 54 T),
Calcium Silicate Block-Type Thermal Insulation (C 344 – 54 T), and
Calcium Silicate Thermal Insulation for Pipes (C 345 – 54 T).

Tentative Method of Test for:

Thermal Conductance and Transmittance of Built-Up Sections by Means of the Guarded Hot Box (C 236 – 54 T),
Thermal Conductivity of Pipe Insulation (C 335 – 54 T),
Mean Specific Heat of Thermal Insulation (C 351 – 54 T), and
Water Vapor Transmission of Materials Used in Building Construction (C 355 – 54 T).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee S-I on Block and Pipe Insulation (P. Losse, chairman) prepared the proposed Tentative Specification for Cellular Glass Thermal Pipe Insulation, referred to previously in the report, and is developing a specification for asbestos block-type thermal insulation for Elevated Temperatures.

The subcommittee is developing methods of test for stress corrosion cracking of austenitic stainless steels attributable to thermal insulation and methods of handleability of thermal insulation.

Subcommittee S-II on Structural Insulating Board (R. R. Sullivan, chairman) is working on a nail head pull through test for building fiberboards, methods of testing insulating roof deck slabs, and insulating formboard. The subcommittee is also developing definitions of structural insulating board and is considering alternate fuel provision for inclusion in the inclined panel flame Test of Methods of Testing Structural Insulating Board Made From Vegetable Fibers (C 209 – 55 T).

Subcommittee S-III on Insulating Cement (E. J. Davis, chairman) prepared the Method of Test for Wet Adhesion of

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Thermal Insulating Cements to Metal, and the definition of finishing cement referred to previously in the report. The subcommittee is considering a method of test for consistency of wet mixed insulating cements and there is under development a specification for mineral wool hydraulic setting thermal insulating and finishing cement.

Subcommittee S-IV on Blanket Insulation (J. M. Barnhart, chairman) prepared the Specification for Mineral Wool Felt Insulation (Industrial Type) for Elevated Temperatures, referred to earlier in the report, and it is developing a specification for batt or blanket type thermal insulation for buildings.

Subcommittee S-V on Loose Fill Insulation (L. A. Barron, chairman) is continuing work on a method of test for determining the density of loose-fill building insulations by the jolting impact method.

Subcommittee T-VI on Thermal Conductivity (F. M. Gavan, chairman) is preparing a method of test for thermal conductivity of insulating materials at low temperatures by means of the Wilkes Calorimeter.

The subcommittee is reviewing the methods of test for Thermal Conductance and Transmittance of Built-Up Sections by Means of the Guarded Hot Box (C 236 – 54 T), Thermal Conductivity of Pipe Insulation (C 335 – 54 T), and Thermal Conductivity of Materials by Means of the Guarded Hot Plate (C 177 – 45).

Subcommittee T-VII on Special Thermal Properties (W. R. Seipt, chairman) is developing methods of test for emissivity at room temperatures, abrasion resistance, and hot surface performance, and it is considering fire resistance tests for thermal insulating materials.

The subcommittee is reviewing the Tentative Method of Test for Mean Specific Heat of Thermal Insulation (C 351 – 54 T).

Subcommittee S-VIII on Dimensional Standards (H. P. Hoopes, chairman) is continuing work on a method of sampling preformed insulation, and a recommended practice for prefabrication of insulation covers for fittings, flanges, and valves.

Subcommittee T-IX on Vapor Transmission (F. A. Joy, chairman) is conducting a series of round-robin tests in accordance with the Tentative Method of Test for Measuring Water Vapor Transmission of Materials in Sheet Form (E 96 – 53 T).

The subcommittee is reviewing the Tentative Method of Test for Water Vapor Transmission of Materials Used in Building Construction (C 355 – 54 T).

Subcommittee S-XI on Reflective Insulation (G. E. Gronemeyer, chairman) is developing definitions relating to reflective insulation.

Subcommittee T-X on Physical Properties (Other Than Permeance) of Coatings Accessory to Thermal Insulation (H. E. Rapp, chairman) is conducting a series of round-robin tests on a method for determining combustibility rates of coatings.

This report has been submitted to letter ballot of the committee, which consists of 91 voting members; 66 members returned ballots, of whom 54 voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

E. R. QUEER,
Chairman.

W. E. GANTZ,
Secretary.

REPORT OF COMMITTEE C-18
ON
NATURAL BUILDING STONES*

Committee C-18 on Natural Building Stones held one meeting during the year: on April 13, 1956, at Washington, D. C. A meeting of the Advisory Subcommittee was held at Washington, D. C., on November 4, 1956. Meetings of subcommittees III on Test Procedures, IV on Specifications, and V on Research were held concurrently with the general meeting of the committee.

Task groups have been set up to work on granite, sandstone, limestone, marble, and slate; programs of work have been initiated and specifications are in varying stages of development.

The officers elected for the ensuing term of two years are as follows:

Chairman, L. W. Currier.
Vice-chairman, T. I. Coe.
Secretary, F. S. Eaton.

The committee consists of 26 members; of whom 5 are classified as producers, 11 as consumers, and 10 as general interest members.

REVISION OF TENTATIVE

The committee recommends that the Tentative Method of Test for Durability of Slate for Roofing (**C 217 – 48 T**)[1] be revised as appended hereto[2] and continued as tentative.

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends that the Tentative Method of Test for Combined Effect of Temperature Cycles and Weak-Salt Solutions on Natural Building Stone (**C 218 – 48 T**), which has stood for more than two years without revision, be continued as tentative.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

Respectfully submitted on behalf of the committee,

L. W. CURRIER,
Chairman.

F. S. EATON,
Secretary.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] 1955 Book of ASTM Standards, Part 3.

[2] This new tentative appears in the 1956 Supplement to Book of ASTM Standards, Part 3.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE C-20
ON
ACOUSTICAL MATERIALS*

Committee C-20 on Acoustical Materials held two meetings during the year: at the University Club in Chicago, Ill., on October 19, 1955, and at ASTM Headquarters, Philadelphia, Pa., on May 3, 1956.

The committee consists of 74 members, of whom 26 are classified as producers, 13 as consumers, 27 as general interest members, and 8 as consulting members.

The officers elected for the ensuing term of two years are as follows:

Chairman, H. A. Leedy.
Vice-Chairman, H. J. Sabine.
Secretary, J. W. Garrison.

NEW TENTATIVE

The committee recommends that the Method of Test for Impedance and Absorption of Acoustical Materials by the Tube Method be accepted for publication as tentative as appended hereto.[1]

This recommendation has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Sound Absorption (H. J. Sabine, chairman) prepared the method of test for impedance and absorption of acoustical materials by the tube method referred to above. Current projects include a reverberation-chamber method for sound absorption measurement and a box method of absorption testing. The subcommittee is following the work of member laboratories on development of a "horn coupler" modification of the impedance tube method, by which specimens larger than the tube diameter may be tested.

Subcommittee II on Flame Resistance (Wallace Waterfall, chairman) has circulated for comment the fourth draft of a test method for flame resistance based on Federal Specification SS-A-118b. A Task Group has been appointed to conduct round robin tests and collect information on the details of panel test equipment now in use. Studies of other existing or proposed methods applicable to acoustical materials, including large and small tunnel methods and the radiant panel method are continuing.

Subcommittee III on Maintenance (W. C. Clark, chairman) is following a project of the National Bureau of Standards to study and measure the soiling characteristics of acoustical materials due to impingement of dirt-laden air. The investigation of suitable test methods for the ability of acoustical materials to withstand repainting and cleaning is continuing.

Subcommittee IV on Application (L. F. Yerges, chairman).—Prior to final adoption of a specification and a test method for acoustical adhesives the sub-committee is reviewing the results of an accelerated test program on aging which is being carried out by a joint task group of Committee C-20 and Committee D-14 on Adhesives. A task group is being set up to investigate mechanical suspension methods of applying acoustical tile.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] This new tentative appears in the 1956 Supplement to Book of ASTM Standards, Part 3.

[2] The letter ballot vote on this recommendation was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee V on Basic Physical Properties (W. A. Jack, chairman).—An article describing apparatus for measuring air-flow resistance of acoustical materials was published in the January, 1956, ASTM Bulletin, for purposes of information and eliciting interest in possible development of a test method. Study is continued on a draft of a method of light reflectance measurement based on the Baumgartner sphere.

This report has been submitted to letter ballot of the committee, which consists of 68 voting members; 39 members returned their ballots, of whom 36 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

H. A. LEEDY,
Chairman.

H. J. SABINE,
Secretary.

REPORT OF COMMITTEE C-21

ON

CERAMIC WHITEWARES AND RELATED PRODUCTS*

Committee C-21 on Ceramic Whitewares and Related Products held two meetings during the year: in Bedford Springs, Pa., on September 29, 1955, and in New York City on April 23, 1956. The Executive Subcommittee met on the same days.

The committee consists of 92 voting members, of whom 52 are classified as producers, 12 as consumers, and 28 as general interest members.

TENTATIVE REVISIONS OF STANDARD

The committee recommends tentative revisions comprising new definitions as appended hereto[1] of the Standard Definitions of Terms Relating to Ceramic Whitewares and Related Products (**C 242 – 55**).[2]

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentative methods be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Methods of:

Sampling Ceramic Whiteware Clays (**C 322 – 53 T**),[2]

Chemical Analysis of Ceramic Whiteware Clays (**C 323 – 53 T**),[2]

Test for Free Moisture in Ceramic Whiteware Clays (**C 324 – 53 T**),[2]

Test for Wet Sieve Analysis of Ceramic Whiteware Clays (**C 325 – 53 T**),[2]

Determination of Drying and Firing Shrinkages of Ceramic Whiteware Clays (**C 326 – 53 T**),[2]

Test for Linear Thermal Expansion of Fired Ceramic Whiteware Materials by the Interferometric Method (**C 327 – 53 T**),[2]

Test for Flexural Properties of Fired Dry-Pressed Whiteware Specimens at Normal Temperatures (**C 328 – 55 T**),[2]

Test for Specific Gravity of Fired Ceramic Whiteware Materials (**C 329 – 53 T**),[2]

Test for Impact Resistance of Ceramic Tableware (**C 368 – 55 T**),[2]

Test for Modulus of Rupture of Fired, Cast or Extruded Whiteware Products (**C 369 – 55 T**),[2]

Test for Moisture Expansion of Fired Whiteware Products (**C 370 – 55 T**),[2]

Test for Sieve Analysis of Nonplastic Pulverized Ceramic Materials (**C 371 – 55 T**),[2]

Test for Linear Thermal Expansion of Fired Whiteware Products by the Dilatometer Method (**C 372 – 55 T**),[2] and

Test for Water Absorption, Bulk Density, Apparent Porosity, and Apparent Specific Gravity of Fired Porous Whiteware Products (**C 373 – 55 T**).[2]

ADOPTION OF TENTATIVE REVISIONS AS STANDARD

The committee recommends that the definitions for "ball clay" and "calcine" which are published as tentative revisions of the Standard Definitions of Terms Relating to Ceramic Whiteware and Similar Products (**C 242 – 55**)[2] be approved for reference to letter ballot of the Society for adoption as standard.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

***Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.**

[1] These new definitions appear in the 1956 Supplement to Book of ASTM Standards, Part 3.

[2] **1955 Book of ASTM Standards, Part 3.**

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature (A. S. Watts, chairman) prepared the definitions appended hereto,[1] and is considering a number of additional definitions, some of which were presented by the Tile Council of America.

Subcommittee II on Tests and Specifications (Van E. Campbell, chairman).—Subsection A1 on Clays (G. W. Phelps, chairman) recommended the adoption as standard of five of the methods referred to previously in this report.

Methods of test are being developed by this committee for hydrometer procedure for particle size determination, dry modulus for ceramic bond clays, and hydrogen ion concentration measurements.

Subsection A3 on Titanate and Zirconate (W. J. Baldwin, chairman).—No general cooperative program was undertaken by this committee during the year, but various committee members carried on some work in an attempt to obtain more consistent electrical values with barium titanate bodies. Considerable laboratory work has been done relative to establishing control tests on barium titanate. This work involves chemical analyses, determination of ceramic and electrical properties, and X-ray examination.

Subsection A4 on Cermets (T. S. Shevlin, chairman) was organized during the September meeting of the committee.

Subsection A5 on Ferrites held its organization meeting in New York City on April 23, 1956.

Subsection C1 on Fundamental Properties (W. C. Mohr, chairman) prepared tests for thermal conductivity of whiteware ceramics, and compressive strength of fired whiteware materials, which are expected to be submitted to the Society through the Administrative Committee on Standards. Eight of the tentative methods of test recommended for adoption as standards were prepared by this subcommittee. Test methods for crazing resistance and translucency are being prepared.

Subcommittee III on Research (H. Z. Schofield, chairman).—The information on measurement of subsieve particle size obtained from round-robin tests is being used by Subcommittee II as an aid in the development of subsieve particle size test procedures for clays and several non-plastic materials.

This report has been submitted to letter ballot of the committee, which consists of 92 voting members; 69 members returned their ballots, of whom 65 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

C. J. KOENIG,
Chairman.

A. J. GITTER,
Secretary.

REPORT OF COMMITTEE C-22
ON
PORCELAIN ENAMEL*

Committee C-22 On Porcelain Enamel held two meetings during the year: on October 5 and 6, 1955, in Pittsburgh, Pa., as guests of the Mellon Institute, and on February 29 and March 1, 1956, in Buffalo, N. Y., during ASTM Committee Week.

During the year two new members have been added to the committee, two have resigned, and one corporate member representative has been replaced. At the present time the committee consists of 43 members of whom 36 are voting members; 20 are classified as producers, 5 as consumers, and 11 as general interest members.

The officers and members-at-large of the Executive Committee elected for the ensuing term of two years are as follows:

Chairman, W. N. Harrison.
First Vice Chairman, D. G. Bennett.
Second Vice Chairman, R. F. Bisbee.
Secretary, G. H. Spencer-Strong.

Members at Large of Executive Subcommittee: A. L. Johnson, F. R. Nagley.

NEW TENTATIVE

The committee recommends that the Method of Test for Thermal Shock Resistance of Porcelain-Enameled Utensils be accepted for publication as tentative as appended hereto.[1]

REVISION OF TENTATIVE

The committee recommends that the Tentative Definitions of Terms Relating to Porcelain Enamel (**C 286–55 T**)[2] be revised by deleting the definition for Porcelain or Vitreous Enamel and adding the following:

> *Porcelain Enamel.*—A substantially vitreous or glassy, inorganic coating bonded to metal by fusion at a temperature above 800 F.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following methods be continued as tentatives without revision:

Tentative Methods of Test for:

Impact Resistance of Porcelain-Enameled Utensils (**C 284 - 51 T**),
Warpage of Porcelain-Enameled Flatware (**C 314 - 53 T**), and
Reflectivity and Coefficient of Scatter of White Porcelain Enamels (**C 347 - 54 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Research (J. J. Canfield, chairman) has seven problems under investigation, the majority of which are of a long-range and continuing nature.

Subcommittee II on Nomenclature (E. E. Howe, chairman) is continuing its review of the Tentative Definitions of

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] This new tentative appears in the 1956 Supplement to Book of ASTM Standards, Part 3.

[2] 1955 Book of ASTM Standards, Part 3.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Terms Relating to Porcelain Enamel (C 286 - 55 T). Because of the demonstrated inadequacies of the existing definition for the term "porcelain enamel," the subcommittee has prepared a more suitable definition as set forth previously in this report.

Subcommittee III on Education (L. S. O'Bannon, chairman) has secured the inclusion of items of educational interest concerning the work of Committee C-22 in some 54 technical publications.

Subcommittee IV on Materials (W. A. Deringer, chairman) is actively working on eight projects. One which is nearly completed is the development of a proposed tentative test method for the measurement of the resistance of porcelain enamels to failure due to torsion.

Subcommittee V on Finished Products (J. C. Richmond, chairman) prepared the new Method of Test for Thermal Shock Resistance of Porcelain-Enameled Utensils which is appended to this report. The subcommittee is presently working on 17 other projects.

This report has been submitted to letter ballot of the committee, which consists of 36 voting members; 30 members returned their ballots, of whom 29 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. N. HARRISON,
Chairman.

G. H. SPENCER-STRONG,
Secretary.

REPORT OF COMMITTEE D-1
ON
PAINT, VARNISH, LACQUER AND RELATED PRODUCTS*

Committee D-1 on Paint, Varnish, Lacquer, and Related Products held two meetings during the year: on June 27 to 29, 1955, in Atlantic City, N. J., in connection with Annual Meeting of the Society, and in Buffalo, N. Y., on February 29, March 1 and 2, 1956, in connection with ASTM Committee Week.

At the June, 1955, meeting, W. W. Cranmer, Industrial Test Laboratory, Philadelphia Naval Ship Yard, presented a paper on "ASTM Committee D-1—Navy Paints." The paper surveyed the types of paint now used by the U. S. Navy and the types expected to be used in the future, and showed the value of the ASTM methods of test prepared by Committee D-1 in controlling quality of paints.

At the February, 1956, meeting, J. A. Stevens, Dow Chemical Co., presented a paper on the "Preparation of Magnesium Surfaces for Painting." For the June, 1956, meeting it is planned to hold a Symposium on the Use of Performance Specifications for Floor Finishes.

During the past year, the new Subcommittee on Putty, Glazing, and Caulking Compounds was organized under the chairmanship of H. L. Kelfer.

Committee D-1 has continued its close cooperation with the Federation of Paint and Varnish Production Clubs through the Joint Federation-ASTM Committee on Paint, Varnish and Lacquer (W. G. Armstrong, chairman). The number of standards which have been jointly approved by both organizations is now 51. Thirteen additional ASTM standards and tentatives are now being considered by the Federation. The organizations are continuing to cooperate in the preparation of specifications for mineral spirits.

Since the last report, the American Standards Association has approved as American Standard the following ASTM specifications and methods of test developed by Committee D-1:

Standard Specifications for:

Raw Linseed Oil (D 234 - 55),
Boiled Linseed Oil (D 260 - 55), and

Tentative Method of Test for:

Tinting Strength of White Pigments (D 332 - 55 T).

The Committee on Intercommittee Relations, composed of D-1 representatives on other ASTM technical committees, is continuing to maintain close contact with those committees whose activities are of interest to Committee D-1. In the last year a representative of Committee D-1 has also been appointed to the Sectional Committee on Electrical Insulating Materials (ASA Project C 59).

During the year the committee approved and amended the section on attendance at meetings of the Regulations Governing Committee D-1.

Chairman Pearce, who was first elected to Committee D-1 in 1921, was given the ASTM Award of Merit by the

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17-22, 1956.

Society at the June, 1955, Annual Meeting.

At the June, 1955, meeting the committee voted unanimously to elect J. F. Broeker to Honorary Membership in Committee D-1.

The officers elected for the ensuing term of two years are as follows:

Chairman, W. T. Pearce.
Vice-Chairman, E. W. Fasig.
Secretary, W. A. Gloger.

Proposed Methods to be Published as Information

The committee recommends for publication as information the following two proposed methods, as appended hereto:[1]

Proposed Methods for Preparation of:

Aluminum and Aluminum-Alloy Surfaces for Painting, and
Magnesium-Alloy Surfaces for Painting.

New Tentatives

The committee recommends that the following nine specifications and methods be accepted for publication as tentative as appended hereto,[2] or as otherwise indicated:

Tentative Specifications for:

Safflower Oil,

Tentative Methods of Test for:

Flash Point of Drying Oils,
Chemical Analysis of White Titanium Pigments,
Abrasion Resistance of Clear Floor Coatings,
Analysis of Polyvinyl Butyral,[3]
Unsaponifiable Matter in Alkyd Resins and Resin Solutions,
Fatty Acid Content of Alkyd Resins and Resin Solutions,
Measurement of Dry Film Thickness of Nonmetallic Coatings of Paint, Varnish, Lacquer, and Related Products Applied on a Nonmagnetic Metal Base, and
Unsaponifiable Content of Tricresyl Phosphate.

Revision of Tentatives

The committee recommends that the following four methods be revised as indicated and continued as tentative:

Tentative Methods of Chemical Analysis of White Pigments (34 – 51 T):[4]

Replace the present procedures for titanium pigments in Sections 32 to 41 by a reference to the new Tentative Methods for Chemical Analysis of White Titanium Dioxide Pigments appended hereto.[2]

Tentative Method of Test for Distillation Range of Lacquer Solvents and Diluents (D 1078 – 49 T):[4]

Revise as appended hereto.[2]

Tentative Method of Test for Nonvolatile Content of Resin Solutions (D 1259 – 53 T):[4]

Revise as appended hereto.[2] The results of the cooperative study of the foil method being added to D 1259 this year are given in Table I.

Tentative Method of Test for Flash Point of Volatile Flammable Materials by Tag Open-Cup Apparatus (D 1310 – 55 T):[4]

A summary of the cooperative test data on the study of this method is appended hereto.[5] The revisions are as follows:

New Section.—Add the following New Section 7 and Note 7, renumbering the present Sections and Note accordingly:

> 7. *Standardization.*—(*a*) Make determinations in triplicate on the flash point of standard para-xylene and of standard isopropyl alcohol which meet specifications set forth in Appendix II. Average these values for each compound. If the difference between the values for these two compounds is less than 15 F (8.5 C) or more than 27 F (16 C), repeat the determinations or obtain fresh standards.

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[1] See pp. 245 and 249.

[2] These new and revised specifications and methods of text appear in the 1956 Supplement to Book of ASTM Standards, Part 4.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 55, p. 445 (1955).

[4] 1955 Book of ASTM Standards, Part 4.

[5] See p. 262.

(*b*) Calculate a correction factor as follows:

Observed Flash of para-xylene = A

Observed Flash of isopropyl alcohol = B

$$X = 92 - A$$

$$Y = 71 - B$$

$$\text{Correction} = \frac{X + Y}{2}$$

Apply this correction to all determinations. Half units in correction shall be discarded.

NOTE 7.—The height of the taper arm is very important. Raising the taper 0.01 in. increases the flash about 2 F. Therefore, if it is suspected that the taper arm has been jarred or bent the apparatus should be recalibrated. Each unit of apparatus should have its calibration checked about once a week if in constant use, or on each occasion of use if used only occasionally.

Note 7.—Renumber as Note 8 and change to read as follows:

NOTE 8.—The calibration procedure provided in this method will cancel out the effect of barometric pressure if calibration and tests are run at same pressure.

Appendix.—Redesignate the present Appendix as Appendix I, and add the following as Appendix II:

SPECIFICATIONS FOR p-XYLENE AND ISOPROPANOL

Specifications for p-Xylene, Flash Point Check Grade:

A2. *p*-Xylene shall conform to the following requirements:

Specific Gravity, 15.56 C/15.56 C......0.860 min, 0.866 max.

Boiling Range......2 C max from start to dry point, when tested by ASTM Method D 850, Test for Distillation of Industrial Aromatic Hydrocarbons, or Method D 1078, Test for Distillation Range of Lacquer Solvents and Diluents. The range shall include the boiling point of pure *p*-xylene, which is 138.35 C (281.03 F).

Purity......95 per cent min, calculated in accordance with ASTM Method D 1016, Test for Determination of Purity from Freezing Points of High-Purity Compounds, from the experimentally determined freezing point, measured by ASTM Method D 1015, Test for Measurement of Freezing Points of High-Purity Compounds for Evaluation of Purity.

Specifications for Isopropanol, Flash Point Check Grade:

A3. Isopropanol shall conform to the following requirements:

Specific Gravity......0.8175 to 0.8185 at

TABLE I.—RESULTS OF COOPERATIVE TESTS BY FOIL METHOD FOR NONVOLATILE CONTENT OF HEAT-REACTIVE RESIN SOLUTIONS.

Laboratory	Amberol ST-137 70 per cent		Uformite F-233, 50 per cent		Bakelite BV-9700A		Varcum 1281-B 80 per cent		Monsanto P-97, 50 per cent		Range Within Laboratory, Average, per cent
	Average, per cent	Range, per cent	Average, per cent	Range, per cent	Average, per cent	Range, per cent	Average, per cent	Range, per cent	Average, per cent	Range, per cent	
Aberdeen	67.6	0.3	54.1	0.7	50.3	1.5	78.0	0.7	47.2	0.5	0.7
Ball Chemical	68.0	0.3	54.0	0.7	51.3	0.8	78.4	0.9	48.5	0.6	0.7
Bakelite	...	...	57.9[a]	0.5	54.0[a]	0.3	81.5[a]	0.6	...	...	0.5
Barrett	68.1	0.3	54.2	0.3	50.4	0.9	79.6	0.2	47.6	0.2	0.4
Cyanamid	67.8[b]	0.8	53.5	0.5	50.4[b]	1.2	77.5	0.4	47.2[b]	1.2	0.8
Hercules	68.5	0.8	53.6	0.7	50.9	0.8	78.1	0.4	47.9	0.7	0.7
Interchemical	69.6	0.3	54.6	0.2	51.5[b]	0.6	79.4	0.2	48.8	0.2	0.3
Rohm & Haas	68.0	0.9	53.3	1.7	50.3	1.0	78.2	0.3	47.7	0.5	0.9
Average of Average Results	68.2	...	53.9	...	50.7	...	78.5	...	47.8	...	...
Range of Average Results	2.1	...	1.1	...	1.2	...	2.1	...	1.6	...	...
Average Range	...	0.5	...	0.8	...	0.9	...	0.5	...	0.6	...

[a] Average of only 3 results; omitted from average result.

[b] Average of the first 6 of the 8 results reported.

20/20 C as determined by means of a calibrated pycnometer.

Distillation Range......shall entirely distill within a 1.0 C range which shall include the temperature 80.4 C as determined by ASTM Method D 1078.

Tentative Revisions of Standards

The committee recommends tentative revisions of the following standards as indicated:

Standard Definitions of Terms Relating to Paint, Varnish, Lacquer, and Related Products (D 16 – 52):[4]

Change the definition of thinner to read as follows:

Thinner.—The portion of a paint, varnish, lacquer, or printing ink, or related product that volatilizes during the drying process.

Standard Methods of Testing Varnishes (D 154 – 53):[4]

Sections 30, 31, and 32.—Change to read as follows:

Acid Value

30. *Reagents:* (*a*) *Titrant* (0.1*N* Potassium Hydroxide in Methyl Alcohol).—Use synthetic methyl alcohol meeting ASTM Specifications D 1152. Standardize using potassium hydrogen phthalate and 1 per cent phenolphthalein indicator.

(*b*) *Neutral Solvent Mixture.*—Mix equal parts of isopropyl alcohol (ASTM Specifications D 770) and industrial toluol (ASTM Specifications D 362). Neutralize, using 0.1*N* KOH as above and 1 per cent phenolphthalein indicator until pink color persists for 1 min.

(*c*) *Indicator* (*1 Per Cent Phenolphthalein*).—Dissolve 1 g of phenolphthalein in 100 ml of methyl alcohol (ASTM Specifications D 1152).

31. *Procedure.*—Weigh in duplicate 3.00 to 5.00 g of varnish into a 500-ml Erlenmeyer flask. Add 100 ml of neutral solvent. Mix until dissolved. Add 1 ml of indicator and titrate with 0.1*N* KOH solution until pink color persists for 1 min.

32. *Calculation.*—Calculate the acid value as the weight in milligrams of potassium hydroxide required to neutralize 1 g of varnish as follows:

$$\text{Acid value} = \frac{AB}{W}$$

where:

A = milliliters of KOH solution required for titration,

B = milligrams of KOH per milliliters of titrant, and

C = weight of the varnish in grams.

Standard Methods of Testing Drying Oils (D 555 – 54):[4]

New Sections.—Add the following new Sections 87 to 90 covering a method of test for heat-bleach of drying oils:

87. *Scope.*—This method covers a procedure for determining the color change of drying oils due to heat.

88. *Definition.*—*Heat-Bleach* of a drying oil is the effect on the color of the oil when it is heated to a specified temperature and held at that temperature for a specified time.

89. *Apparatus:* (*a*) *Beaker.*—A 250-ml beaker, Griffin low-form, of heat-resistant glass.[a]

(*b*) *Ring Stand.*—Ring stand with asbestos wire pad.

(*c*) *Heater.*—A Fisher high-temperature gas burner or other suitable gas burner.

(*d*) *Thermometer.*—An ASTM Partial Immersion Thermometer No. 3 F having a range of +20 to +760 F and conforming to the requirements in ASTM Specifications E 1.

(*e*) *Color Standards.*—A set of Gardner 1933 permanent color standards, as described in Section 5 of the Standard Methods of Testing Varnishes (ASTM Designation: D 154).

90. *Procedure.*—Place 100 ml of the oil sample in the 250 ml beaker. Immerse the thermometer in the center of the oil so that the end of the bulb is from $\frac{1}{4}$ to $\frac{5}{16}$ in. from the bottom of the beaker. Heat to 550 F in 5 to 10 min, and hold at this temperature ±5 F for 5 min. Remove the beaker from the stand and allow to cool to room temperature. Determine the color of the sample according to Sections 5 to 7 of Standard Methods D 154.

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[a] Pyrex glass is suitable for this purpose.

Revision of Standard and Reversion to Tentative

The committee recommends that the following standard be revised as appended hereto[2] and reverted to tentative status:

Standard Specifications for Raw Soybean Oil (**D 124 – 48**).[4]

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following ten tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Methods of Test for:

No-Dirt-Retention Time of Traffic Paint (**D 1297 – 53 T**),[4]
Phthalic Anhydride Content of Alkyd Resins and Esters Containing Other Dibasic Acids (Gravimetric) (**D 1306 – 54 T**),[4]
Phthalic Anhydride Content of Alkyd Resins and Esters Containing Other Dibasic Acids (Spectrophotometric) (**D 1307 – 54 T**),[4]
Settling Properties of Traffic Paints During Storage (**D 1309 – 54 T**),[4]
Apparent Free Phenols in Synthetic Phenolic Resins or Solutions Used for Coating Purposes (**D 1312 – 54 T**),[4]
Nonvolatile Matter in Lacquer Solvents and Diluents (**D 1353 – 54 T**),[4]

Tentative Methods of Testing:

Varnishes (**D 154 – 53 T**),[4] to be added to Standard D 154 – 53

Tentative Method for:

Evaluating Degree of Blistering of Paints (**D 714 – 54 T**),[4]

Tentative Recommended Practice for:

Conducting Exterior Exposure Tests of Paints on Wood (**D 1006 – 51 T**),[4] and

Tentative Specifications for

Asphalt-Base Emulsions for Use as Protective Coatings for Metal (**D 1187 – 51 T**).[4]

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions in the following five standards, as appended hereto[2] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Specifications for:

Ethylene Glycol Monobutyl Ether (**D 330 – 35**),[4]
Ethylene Glycol Monoethyl Ether (**D 331 – 35**),[4]
Acetate Ester of Ethylene Glycol Monoethyl Ether (95 to 96 per cent Grade) (**D 343 – 35**),[4]
Tricresyl Phosphate (**D 363 – 49**),[4] and
Methyl Ethyl Ketone (**D 740 – 46**).[4]

Committee D-1 concurs in the proposed revision of the Standard Method of Test for Flash Paint by Tag Closed Tester (D 56 – 52) being recommended by Committee D-2 on Petroleum Products and Lubricants.[6]

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following ten tentatives be continued without revision:

Tentative Specifications for:

Heavy Petroleum Spirits (Heavy Mineral Spirits) (**D 965 – 48 T**),

Tentative Methods of Test for:

Ester Value of Tricresyl Phosphate (**D 268 – 49 T**),
Kauri-Butanol Value of Hydrocarbon Solvents (**D 1133 – 54 T**),
Purity of Acetone and Methyl Ethyl Ketone (**D 1154 – 54 T**),
Temperature-Change Resistance of Clear Nitrocellulose Lacquer Films Applied to Wood (**D 1211 – 52 T**),
Nonvolatile Content of Resin Solutions (**D 1259 – 53 T**),
Effect of Household Staining Agents on Applied Nitrocellulose Clear and Pigmented Finishes (**D 1308 – 54 T**),
Viscosity Reduction Power of Hydrocarbon Solvents (**D 1311 – 54 T**),

Tentative Methods of:

Testing Shellac (**D 29 – 54 T**), and

Tentative Recommended Practice for:

Operating Light and Water Exposure Apparatus (Carbon-Arc Type) for Testing Paint, Varnish, Lacquer, and Related Products (**D 822 – 46 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[7]

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[6] See p. 274.

[7] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Drying Oils (D. S. Bolley, chairman) prepared the proposed Tentative Specifications for Safflower Oil and the proposed Tentative Method of Test for Flash Point of Drying Oils, referred to earlier in the report, as well as the tentative revision of Methods D 555 to include a proposed method for determination of heat-bleach color of drying oils, and the proposed tentative revision of Specifications for Raw Soybean Oil (D 124). The subcommittee is preparing a proposed procedure for sampling drying oils, proposed methods of test for fatty acids, and a revision of Specifications for Raw Linseed Oil (D 234), in which the foots requirements will be replaced with a closely defined break test.

Subcommittee III on Bituminous Emulsions (R. H. Cubberley, chairman) recommended that the Tentative Specifications for Asphalt-Base Emulsions for Use as Protective Coatings for Metal (D 1187 - 51 T) be adopted as standard.

Subcommittee IV on Traffic Paint (W. W. Burr, chairman) recommended the adoption as standard of the Tentative Method of Test for No-Dirt-Retention Time of Traffic Paint (D 1297 - 53 T), and the Tentative Method of Test for Settling Properties of Traffic Paint During Storage (D 1309 - 54 T), with an editorial revision in the scope of the latter method limiting it to unbeaded traffic paints.

Subcommittee V on Volatile Solvents for Organic Protective Coatings (M. B. Chittick, chairman) is continuing its work on distillation procedures and studies of evaporation rates.

Subcommittee VI on Definitions (G. G. Sward, chairman) prepared the proposed tentative revision of the Standard Definitions of Terms Relating to Paint, Varnish, Lacquer, and Related Products (D 16) consisting of a change in the definition for Volatile Thinner.

Subcommittee VII on Accelerated Tests for Protective Coatings (H. D. Pharo, chairman) recommended adoption as standard of the Tentative Method for Evaluating Degree of Blistering of Paints (D 714 - 54 T), and Tentative Recommended Practice for Conducting Exterior Exposure Test of Paints on Wood (D 1006 - 51 T). The subcommittee is revising the Tentative Recommended Practice for Operating Light and Water Exposure Apparatus (Carbon-Arc Type) for Testing Paint, Varnish, Lacquer, and Related Products (D 822 - 54 T) in order to have it conform with the Tentative Recommended Practice for Operation of Light- and Water-Exposure Apparatus (Carbon-Arc Type) for Artificial Weathering Test (E 42 - 55 T). The subcommittee is cooperating with Subcommittee XXV on Cellulosic Coatings and Related Materials on broadening the scope of the Tentative Method of Test for Effect of Household Staining Agents on Applied Nitrocellulose Clear and Pigmented Finishes (D 1308 - 54 T), to include all household chemicals and all organic coatings. The staining of transportation finishes, the water fog test, and the yellowing of interior finishes are other problems on which the subcommittee is working.

Subcommittee VIII on Methods of Chemical Analysis of Paint Materials (W. H. Madson, chairman) prepared the proposed Tentative Methods for Chemical Analysis of White Titanium Pigments. It is developing a method for determination of soluble salts of pigments by conductivity methods, and a revision of the Standard Methods of Chemical Analysis of White Linseed Oil Paints (D 215), to provide for the determination of soluble salt content of combinations of pigments. Revisions of the Standard Method of Chemical Analysis of Dry Mercuric Oxide (D 284), are nearly completed.

Subcommittee IX on Varnish (J. C. Weaver, chairman) prepared the proposed Tentative Method of Test for Abrasion Resistance of Clear Floor Coatings, and the proposed tentative revision relating to the acid number test in the Standard Methods of Testing Varnishes (D 154 - 53). The subcommittee recommended the adoption as standard of the Tentative Methods of Testing Varnishes (D 154 - 53 T). The subcommittee is working on correction of certain tube standards in the Gardner system for determining color of transparent liquids, the development of methods for skinning tests, and for determination of rosin acids content in varnishes, specifications for driers, and the redraft of a number of tests which apply to varnishes covered by Federal Methods of Test TT-P-141.

Subcommittee X on Optical Properties (M. P. Morse, chairman) has as current activities the preparation of three draft methods for measurement of color differences using the General Electric Spectrophotometer, the Colormaster, and the Color Eye Colorimeter, a method for measuring gloss of clear finishes on wood, tables to facilitate the calculations of spreading rates from reflectance data of paints, improved procedures for preparing uniform films and density determinations, and a revision of Tentative Method of Test for Tinting Strength of White Pigments (D 332 - 55 T) to include the Hoover Muller as an alternate method of dispersion. The subcommittee will also undertake the development of a satisfactory light source and conditions for visual color matching, and procedures for expressing color in the Munsell color system.

Subcommittee XI on Resins (C. F. Pickett, chairman) recommended the adoption as standard of the Tentative Methods of Test for Phthalic Anhydride Content of Alkyd Resins and Esters Containing Other Dibasic Acids (Gravimetric) (D 1306 - 54 T), for Phthalic Anhydride Content of Alkyd Resins and Esters Containing Other Dibasic Acids (Spectrophotometric) (D 1307 - 54 T), and for Apparent Free Phenols in Synthetic Phenolic Resins or Solutions Used for Coating Purposes (D 1312 - 54 T). The subcommittee prepared the proposed Tentative Methods of Test for Unsaponifiable Content of Alkyd Resins, and for Total Fatty Acid Content of Alkyd Resins. The revised Test for Nonvolatile Content of Resin Solutions (D 1259) is a combination method applicable to both heat-stable and heat-unstable resins. The results of the cooperative study of the foil method are given in Table I.

The subcommittee also prepared the proposed Tentative Method for Analysis of Polyvinyl Butyral which was published as information in 1955. It is working on a melt procedure for use with heat-reactive resins, determination of viscosity of resin solutions using viscosity tubes, the determination of pentaerythritol, glycerol and ethylene glycol in alkyds, the determination of melamine in resin solutions, and the determination of color as a result of ester formation from the reaction of glycerine and phthalic anhydride.

Subcommittee XII on Latex and Emulsion Paints (P. T. Howard, chairman) is continuing its cooperative studies on washability and scrubbability, efflorescence, freeze-thaw resistance, and exterior exposure tests. Bacterial contamination in the package and mildew growth on dried films are under study by a new working group on microbiological tests. A new group on coalescence was formed to study the problem of poor film formation, particularly during application to cold surfaces.

Subcommittee XIII on Shellac (C. C. Hartman, chairman) will undertake revision of the values for per cent insoluble matter of several grades of orange shellac listed in the Standard Specifications for Orange Shellac and Other Lacs (D 237), and revision of the method for determination of acid number of bleached lac and bleached lac varnish in Tentative Methods of Testing Shellac (D 29 – 54 T). Further work will be done on determinations of the bleach index and bleachability of seed lac, and on nonvolatile matter soluble in cold alcohol. In its work on the International Committee on Lac (ISO/TC 50) the subcommittee has recommended approval by the American Standards Association of specifications for seed lac, shellac, and bleached lac.

Subcommittee XV on Specifications for Pigments, Dry and in Oil (C. L. Crockett, chairman) is working on revisions of the Standard Specifications for Copper Phthalocyanine Blue (D 963 – 49), and on revisions of the leafing tests and sieve requirements of the Standard Methods of Sampling and Testing Aluminum Powder and Paste (D 480 – 51), and Standard Specifications for Aluminum Pigments, Powder and Paste, for Paints (D 962 – 49).

Subcommittee XVI on Printing Inks (M. C. Rogers, chairman) has in preparation a proposed tentative method for determination of specific gravity of both paste and free-flowing printing inks.

Subcommittee XVII on Flash Point (A. L. Brown, chairman) prepared the proposed revision of the Tentative Method of Test for Flash Point of Volatile, Flammable Materials by Tag Open-Cup Apparatus (D 1310 – 55 T). The subcommittee will investigate the determination of flash point of nonviscous materials in the range of 0 F to 60 F, and of viscous materials in the range of 60 F to 100 F.

Subcommittee XVIII on Physical Properties of Materials (E. J. Dunn, Jr., chairman) prepared the proposed Tentative Method for Measurement of Dry Film Thickness of Nonmetallic Coatings of Paint, Varnish, Lacquer, and Related Products Applied to a Nonmagnetic Metal Base, referred to earlier in the report. The subcommittee is working on a proposed tentative method for determination of hardness of films using the Tukon tester, a proposed tentative method of test for oil absorption by the Gardner-Coleman procedure, application of uniform films, hardness of films using the Sward-Rocker and the Pfund type instrument, water vapor permeability, rheological properties, density and adhesion, and a method for measurement of film thickness of finishes on wood.

Subcommittee XIX on Putty, Glazing and Caulking Compounds (H. L. Kelfer, chairman) was organized during the year. The following nine tests have been chosen as those which should adequately evaluate quality by performance standards only: adhesion, flexibility, initial and ultimate set, staining into substrate, ultimate appearance, surface dry, slump, workability, and package stability. Working groups have been formed to work on the above test procedures.

Subcommittee XXV on Cellulosic Coatings and Related Materials (N. C. Schultze, chairman) prepared the proposed Tentative Method of Test for Unsaponifiable Content of Tricresyl Phosphate, and the proposed revision of the Tentative Method of Test for Distillation Range of Lacquer Solvents and Diluents (D 1078 – 49 T). It recommended the adoption as standard of the Tentative Method of Test for Nonvolatile Matter in Lacquer Solvents and Diluents (D 1353 – 54 T), and the adoption of revisions in Standard Specifications D 330 – 35, D 331 – 35, D 343 – 35, D 363 – 49, and D 740 – 46.

A joint group has been set up with Subcommittee VII on Accelerated Tests for Protective Coatings to revise the scope of Tentative Method of Test for Effect of Household Staining Agents on Applied Nitrocellulose Clear and Pigmented Finishes (D 1308 – 54 T) to include all organic coatings. Revision of the Tentative Method of Test for Temperature-Change Resistance of Clear Nitrocellulose Lacquer Films Applied to Wood (D 1211 – 52 T) is under way, and work has been started on a test method for determining perspiration resistance of lacquers, and the modernization of solvent specifications.

Subcommittee XXIX on Painting of Metals (A. J. Eickhoff, chairman) prepared the two proposed methods which are referred to previously in the report for publication as information only.

In its work on classification of ferrous surfaces for painting, the subcommittee is considering the use of stereoscopic color transparencies as a means of showing conditions of metal surfaces.

This report has been submitted to letter ballot of the committee, which consists of 364 voting members; 173 members returned their ballots, of whom 158 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. T. PEARCE,
Chairman.

W. A. GLOGER,
Secretary.

APPENDIX I

PROPOSED METHODS FOR PREPARATION OF ALUMINUM AND ALUMINUM-ALLOY SURFACES FOR PAINTING[1, 2]

These are proposed methods and are published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. (*a*) These methods cover four types of treatment for preparation of aluminum and aluminum-alloy surfaces for painting, as follows:

Type A. Solvent Cleaning.
Type B. Chemical Treatments.
Type C. Anodic Treatments.
Type D. Mechanical Treatments.

These four types cover a number of procedures, as described herein.

(*b*) Variations in surface treatment produce end conditions which differ, and which do not necessarily yield identical results when paints are applied. Service conditions will dictate the type of surface preparation that should be selected, although the quality produced by any individual method may vary with different alloys.

Type A. Solvent Cleaning

2. Solvent cleaning does not disturb the natural oxide film on the metal and may prove adequate for some applications, such as ambient indoor or very mild service conditions. Three methods may be employed, as follows:

(*a*) *Method 1. Manual Swabbing or Dip-Washing*, with a solvent such as mineral spirits or high-flash solvent naphtha. With this method it is extremely difficult to prevent accumulation of contaminants on the swab or in the solvent. This method is only recommended when other treatments are impractical.

(*b*) *Method 2. Solvent Spray Cleaning*, according to Method 1, Procedure A of the Method of Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products (ASTM Designation: D 609).[3]

(*c*) *Method 3. Vapor Degreasing*, in special equipment employing trichloroethylene vapor, according to Method 2, Procedure A of Method D 609.

Type B. Chemical Treatments (see Note 1)

3. (*a*) *Method 1. Alkaline Cleaners.*—Alkaline solutions, such as caustic soda, etch the metal, thus destroying the natural oxide film. They are followed by an acid treatment, preferably nitric acid or phosphoric acid. They shall not be

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[1] These proposed methods are under the jurisdiction of the ASTM Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

Publication as information only requires letter ballot of the appropriate subcommittee of Committee D-1, and approval of the chairman and secretary of Committee D-1. Letter ballot of Committee D-1 as a whole is not required.

[2] Published as information, June, 1956.

[3] 1955 Book of ASTM Standards, Part 4.

used on assembled structures. Inhibited alkaline cleaners are sometimes employed as a pretreatment to remove grease and oil prior to an acid treatment. Inhibited alkaline cleaners do not etch the surface. They are not generally recommended unless followed by a conversion treatment, such as described in Methods 4, 5, 6, or 7.

(*b*) *Method 2. Sulfuric-Chromic Acid Etch.*—This treatment provides a clean and uniform surface without undue etching, and is effective for removing oil and water stains and any film formed during heat-treatment. The etching solution is prepared by adding 1 gal of concentrated sulfuric acid and 45 oz of chromic acid to 9 gal of water. It is used at a temperature of 160 to 180 F (depending on the alloy and the amount of film) for about 5 min and is followed by a water rinse. This treatment produces a passive surface suitable for painting under mild to intermediate exposure conditions and where clear finishes are to be applied.

(*c*) *Method 3. Alcoholic Phosphoric Acid Cleaners.*—This treatment involves the use of an aqueous solution of phosphoric acid (10 to 15 per cent by volume) with alcohol or other organic solvents, together with wetting agents, emulsifying agents, etc. The solution may be applied by swabbing or dipping at room temperature (70 to 90 F), and should be allowed to remain on the surface for several minutes, followed by thorough rinsing with clean water. A very thin phosphate film is formed which tends to protect the metal and promote paint adhesion under mild to intermediate exposure conditions (see Note 2).

(*d*) *Method 4. Crystalline Phosphate Treatment.*—This surface-coating method consists in reacting the aluminum surface in a zinc-acid-phosphate solution containing oxidizing agents and other salts for accelerating the coating action. The aluminum surface is converted to a finely crystalline, phosphate coating of the proper texture adapted to inhibit corrosion and increase the adherence and durability of any applied paint film. It is recommended for product finishes. The phosphate coating process may be carried out by immersion or spray application. The aluminum surface is converted to the phosphate coating by immersion in the processing solution for 30 sec to 4 min at 125 to 140 F, or by spraying the solution for 10 sec to 2 min at 125 to 160 F (see Notes 3 and 4).

(*e*) *Method 5. Amorphous Phosphate Treatment.*—This process comprises treatment of clean aluminum surfaces in a warm (95 to 130 F) aqueous solution comprising phosphoric, chromic, and hydrofluoric acids or a solution thereof. The aluminum surface is converted to a thin, adherent, amorphous coating, iridescent green to gray-green in color, depending upon the aluminum alloy treated, which inhibits corrosion and increases the adherence and durability of applied paint films. This method is recommended for use under the more severe conditions of service, and for product finishes. The coating process may be carried out by immersion or spray application. The time of treatment will vary from 15 to 45 sec for the spray process, and from 30 sec to 3 min for the immersion application (see Notes 3 and 5).

(*f*) *Method 6. Carbonate Chromate Treatment.*—This process comprises treatment in a hot (180 to 190 F) dilute solution of sodium carbonate and potassium chromate for periods from 2 to 20 min, followed by a water rinse. The surface is then given a final treatment in hot 5 per cent potassium dichromate solution, followed by a final rinse. The treatment produces a thin, adherent, conversion coating on the surface, in creasing the corrosion resistance of the metal and promoting paint adhesion.

This method is recommended for use under the more severe conditions of service and for product finishes (see Notes 3 and 5).

(*g*) *Method 7. Amorphous Chromate Treatment.*—This process comprises treatment of clean aluminum surfaces in aqueous chromic acid solutions containing suitable accelerating agents such as fluoride-containing materials. The aluminum surface is converted to an adherent, amorphous, mixed metallic oxide coating, iridescent golden to light-brown in color, which increases the corrosion-resistance and the adherence and durability of any applied paint film. This method is recommended for use under the more severe conditions of service and for product finishes. The coating process may be carried out by immersion, spray, or brush application, at room temperature (70 to 90 F), in from 15 sec to 5 min contact time (see Notes 3 and 5).

(*h*) *Method 8. Acid-Bound Resinous Treatment* (see Note 6).—This surface treatment involves the use of a suitably applied acid-bound resinous film of approximately 0.3 to 0.5 mils thickness. The treatment is based on three primary components: a hydroxyl-containing resin; a pigment capable of reacting with the resin and an acid; and an acid capable of insolubilizing the resin by reacting with the resin, the pigment, and the metal substrate. The aluminum surface should be prepared by Methods 1, 2, or 3 of Type A (see Section 2) prior to the application of this treatment. The film may be applied by brush, spray, or dip. Under normal conditions it should dry sufficiently for recoating within 30 min; and within 8 hr it should not be softened by organic solvents commonly used in paint coatings. The film has good adhesion to the metal substrate and promotes good adhesion of most subsequent organic coatings to itself. This method is recommended for severe service conditions, particularly on fabricated structures, either in the shop or in the field.

Type C. Anodic Treatments (see Note 7)

4. Anodic treatment in either chromic or sulfuric acid electrolyte will provide a protective and inert oxide coating which increases the corrosion-resistance of the metal and promotes paint adhesion. It is recommended where maximum corrosion-resistance by the treatment itself is desired. Anodic treatments should not be used on assemblies of dissimilar metals. The two procedures are as follows:

(*a*) *Method 1. Sulfuric Acid Anodic.*—This treatment consists in making the part the anode in a 15 per cent sulfuric acid electrolyte (by weight) and applying current at a potential of about 16 to 18 v and a current density of 12 amp per sq ft until desired thickness of coating is obtained. After rinsing in cold water, the coating is sealed for 10 min by treatment in a boiling solution of 5 per cent potassium dichromate. This method is not recommended where danger of acid entrapment is encountered.

(*b*) *Method 2. Chromic Acid Anodic.*—This treatment consists in making the part the anode in a 10 per cent (by weight) chromic acid electrolyte at a temperature of 100 F and applying a potential of 40 v for a period of 30 to 60 min. After first rinsing in cold water, the coating is finally rinsed in water at 150 F minimum, and dried; an optional final sealing treatment to obtain maximum protection is obtained by immersion in a solution containing 0.01 g chromic acid per liter of water at a temperature of 208 to 212 F for 5 min.

Type D. Mechanical Treatments

5. (*a*) *Method 1. Hand or Power Wire-Brushing,* or other abrasive treatment.

Wire-brushing, either by hand or power, roughens the surface of the metal and mechanically improves the anchorage for superimposed paint films. The disadvantages are that the natural oxide film on the metal is disrupted and oil or grease films and other foreign matter are not completely removed.

(*b*) *Method 2. Sandblasting*, where employed on aluminum or its alloys, must be carried out at relatively low pressures and with a fine silica sand. It roughens the surface and mechanically improves the anchorage for paint films, but destroys the natural oxide film on the metal. Where used, it should be followed by an inhibitive chemical treatment.

EXPLANATORY NOTES

NOTE 1.—Materials and procedures employed in these methods of treatment are available from a number of sources as proprietary compounds or methods. Selection may be made from available sources.

NOTE 2.—U. S. Military Specification MIL-M-10578A describes a treatment of this type.

NOTE 3.—Before applying the treatments according to Methods 4, 5, 6, and 7 of Type B, the aluminum surfaces should be freed of grease, oil, or other foreign material by means of the procedure described in Method 3 of Type A, Method 1 of Type B, or any other suitable method.

NOTE 4.—This treatment complies with the requirements of U. S. Military Specification MIL-C-490.

NOTE 5.—Most of the treatments conforming to Methods 5, 6, and 7 of Type B comply with the requirements of U. S. Military Specification MIL-C-5541.

NOTE 6.—Materials meeting the requirements of U. S. Military Specification MIL-P-15328 may be used to apply Method 8 of Type B.

NOTE 7.—Treatments conforming to Methods 1 and 2 of Type C comply with the requirements of U. S. Military Specification MIL-A-8625 (ASG).

APPENDIX II

PROPOSED METHODS FOR PREPARATION OF MAGNESIUM-ALLOY SURFACES FOR PAINTING[1, 2, 3]

These are proposed methods and are published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. These methods cover two classes of treatment for preparation of magnesium-alloy surfaces for painting, as follows:

Class I.—Chemical Treatments.
Class II.—Anodic Treatments.

In general, the latter treatments are the more protective of the two classes. Mechanical (abrasive) treatments, solvent cleaning, alkaline solution treatments, and acid pickles not resulting in protective conversion coatings are suitable preliminary treatments only for metal to be exposed under mildly corrosive (indoor) exposures. When a high degree of corrosion protection and paint adhesion are desired, as in many outdoor environments, surface preparation by one of the above conversion-coat classes is necessary (Note 1).

NOTE 1: *Testing of Coatings.*—Quality control tests of coatings are frequently desirable, and these generally consist of exposures, with or without paint, to salt spray, humidity, or natural environments, with suitable procedures for assessing the degree of breakdown suffered after fixed time intervals. It is recommended that quality control tests of coatings shall be made as far as possible with high-purity material (for example AZ31A alloy)[4], the inherent corrosion rate of which is relatively consistent from batch to batch) and that precautions shall be taken to remove surface contamination before coatings are applied. Such contamination shall be removed by acid pickling to a depth of at least 0.001 in. per side.

PRELIMINARY TREATMENT OF SURFACES

Procedure

2. Certain anodic treatments simultaneously produce conversion coatings on, and remove contamination from,

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[1] These methods are under the jurisdiction of the ASTM Committee D-1 on Paint, Varnish, Lacquer, and Related Products. Publication for information only requires letter ballot of the appropriate subcommittee of Committee D-1, and approval of the chairman and secretary of Committee D-1. Letter ballot of Committee D-1 as a whole is not required.

[2] Published as information June, 1956.

[3] In publishing this recommended practice, the American Society for Testing Materials neither undertakes to protect anyone utilizing them against liability for infringement of Letters Patent, nor assumes any such liability; such publication should not be construed as a recommendation or an endorsement by the Society of any patented or proprietary process that may be involved.

Inasmuch as no control is exercised by the American Society for Testing Materials over the manner in which the methods herein described are used, no guarantee is issued by the Society that results substantially as described or implied herein will be obtained.

[4] For information concerning magnesium and aluminum alloys, see ASTM Specifications B 80, B 90, B 91, B 93, B 107, B 178, and B 209 covering these alloys, in the section on Aluminum and Magnesium and Their Alloys, 1955 Book of ASTM Standards, Part 2. See also, in Part 2, the Recommended Practices for Codification of Light Metals and Alloys, Cast and Wrought (ASTM Designation: B 275).

magnesium-alloy surfaces. In general, however, conversion coatings shall be applied only to surfaces previously freed from all contamination, including oxide, rolling-scale, corrosion product, burned-on drawing and forming lubricant, and the contamination introduced by blast cleaning and fabrication operations. Contamination in or under surface conversion coatings seriously reduces their protective values (Note 2). For the removal of tenacious surface contamination, such as rolling-scale or casting skin, an acid pickle to dissolve some of the actual surface is essential. When organic contamination, such as grease or oil, is also present, an initial degreasing operation in solvent or in an alkaline degreasing solution is usually necessary to allow the subsequent acid to wet the surface. These matters are discussed in more detail under the headings of the specific cleaners or treatments (Note 3), as follows:

(*a*) *Alkaline Cleaners.*—Oil, grease, and old (but not baked) chrome-pickle coatings are readily removed by most commercially available heavy-duty alkaline cleaners; but such cleaners are not suitable for removing oxide and the like, for which purpose acid pickles, preceded by alkaline cleaners, shall be used. Graphite lubricant and also baked chrome-pickle coatings shall be removed by a solution conforming to the following composition:

Caustic soda (NaOH)	12 oz
Wetting agent	0.1 oz
Water (Note 4)	1 gal

The parts shall be soaked in the above cleaner for 10 to 20 min at boiling-point, and a treatment shall follow either in the chromic acid-nitrate pickle described under Paragraph (*b*), Item (*3*) or, for parts machined to fine tolerances, in the chromic acid solution described under Paragraph (*b*), Item (*1*). After alkaline cleaning, rinsing in water shall be extremely thorough. Alkaline cleaners may be held in plain steel tanks.

(*b*) *Acid Cleaners* (Note 1).—Acid pickling removes mill-scale, oxide, corrosion product, and the like, and shall be used as a preliminary treatment for surface conversion coatings when the highest degrees of surface uniformity and protective values are required. Acid cleaners are as follows:

(*1*) *For Sand and Permanent Mold Castings:*

Nitric - Sulfuric Acid Solution.—A solution of 8 per cent by volume of concentrated nitric acid (HNO_3) plus 2 per cent by volume of concentrated sulfuric acid (H_2SO_4) in water (see Note 1(*a*)) shall be used at 70 to 90 F as a preliminary treatment for new sand castings and to remove the surface-contaminating effects of blast cleaning. The time of immersion shall be for about 10 to 15 sec, or until 0.002 in. per surface is removed. The solution may be held in ceramic, rubber, synthetic rubber, or vinyl-lined tanks.

Chromic Acid Solution.—A boiling 20 per cent by weight solution of chromic anhydride (CrO_3) in water shall be used to remove old chemical and anodic treatments, corrosion product, and oxide layers, without significant dissolution of metal and hence without changing the dimensions of machined parts. Immersion time varies from 1 to 5 min, depending upon the condition of the surface. The solution may be held in lead-lined steel or ASTM alloy No. 990A or its Aluminum Association equivalent, alloy No. 1100 aluminum tanks.

(*2*) *For Die Castings:*

Chromic - Nitric - Hydrofluoric Acid Solution.—This solution is used to produce a smut-free surface on die-castings, without violent attack of the metal. The solution shall conform to the following composition:

Chromic acid (CrO_3)	37.5 oz
Hydrofluoric acid (60 per cent HF)	1 fl oz
Nitric acid (70 per cent HNO_3)	3.25 fl oz
Water (Note 4)	to 1 gal

The parts shall be immersed in the above solution at 70 to 90 F for 30 sec to 2 min, or until a bright, clean surface is obtained. The solution may be held in tanks lined with synthetic rubber or vinyl-base materials.

(3) For Wrought Products:

Acetic Acid - Nitrate Solution.—This solution rapidly removes surface contamination to 0.001 in., and shall be used for wrought parts subsequently to be finished for the maximum protective value. The solution shall conform to the following composition:

Glacial acetic acid	25.5 fl oz
Sodium nitrate ($NaNO_3$)	6.6 oz
Water (Note 4)	to 1 gal

The parts shall be immersed in the above solution at 70 to 90 F for 30 sec upwards, or until a bright, clean surface is obtained. When heavy surface contamination, such as hot-rolled mill-scale is to be removed, immersion times shall be sufficient to remove at least 0.001 in. per surface. The solution may be held in No. 990A aluminum, ceramic, or rubber-lined tanks.

Chromic Acid - Nitrate Solution.—This solution shall be used following the use of the method described in Paragraph (*a*) for the removal of burned-on graphite lubricants from hot-formed parts. The solution shall conform to the following composition:

Chromic acid (CrO_3)	1½ lb
Sodium nitrate ($NaNO_3$)	2 oz
Water (Note 4)	to 1 gal

The parts shall be immersed in the above solution at 70 to 90 F for approximately 5 min, for the removal of graphite.

Abnormally slow reaction in the above solution indicates that it is depleted and that the pH has risen to 1.7 or higher. The solution shall be revivified by the addition of chromic acid to bring the pH back to 0.5 to 0.7. No more than four revivifications shall be attempted. The solution may be held in ceramic, No. 990A aluminum, 18-8 stainless steel, or synthetic rubber-lined tanks.

NOTE 2: *Removal of Contamination by Welding Fluxes.*—When a part to be painted has been welded by an operation involving the use of flux, such flux shall be removed before the subjection of the part to any surface preparation process. Such removal shall be made immediately by immersing the part in hot water with scrubbing, and finally by immersing it for 1 hr in a boiling 5 per cent solution of sodium dichromate, after which the part shall be well rinsed.

NOTE 3: *Suspension of Articles for Treatment.*—The use of magnesium alloy suspension wires is preferred for use in acid pickles in order to avoid objectionable contamination of the solutions through dissolution of the wire materials. Heavy metal contamination, particularly of copper, may deposit on the magnesium surface and lead to seriously reduced corrosion resistance. Copper suspension wires in the hot dichromate solutions are not objectionable.

NOTE 4: *Quality of Water.*—In the preparation and make-up of acid pickles, dichromate solutions, and hot-water rinses, precautions shall be taken against the use of water contaminated with heavy-metal impurities, or excessive chlorides or sulfates. No upper limits can be specified at this stage for soluble impurities in the water, but powdering of coatings and poor resistance to corrosion are known to result from the use of contaminated water. Thus, when a choice exists, water from steam condensate or ion-exchange-treated water shall be employed in preference to well water or hard tap water.

SURFACE PREPARATION PROCEDURES—CHEMICAL

Class I, Type I (Chrome Pickle)[5]

3. (*a*) *Scope.*—Class I, Type I treatment is applicable to all forms and alloys of magnesium except certain special alloys containing silver, but since it may remove as much as 0.0006 in. of metal per surface, it shall not be used on parts machined to fine tolerances. When properly applied, the process constitutes a good paint base, but rigid control is required at each step. The treatment is applicable to magnesium alloy containing inserts of, or attached to, other metals.

(*b*) *Procedure.*—For wrought parts the bath shall conform to the following composition:

Sodium dichromate ($Na_2Cr_2O_7 \cdot 2H_2O$)	1.5 lb
Nitric acid (HNO_3) (sp gr 1.42)	1.5 pt
Water (Note 4)	to 1 gal

For die-, sand- and permanent-mold castings the solution shall conform to the following composition:

Sodium dichromate ($Na_2Cr_2O_7 \cdot 2H_2O$)	1.5 lb
Nitric acid (HNO_3) (sp gr 1.42)	1.5 pt
Sodium, potassium, or ammonium acid fluoride ($NaHF_2$, KHF_2, or NH_4HF_2)	2 oz
Water (Note 4)	to 1 gal

For wrought products, sand, and permanent-mold castings the above solutions shall be operated at 70 to 90 F, and the immersion times shall be from 1 to 2 min, the necessary time increasing with use of the solution. For die-castings the parts shall be given a 15 to 30 sec dip in water at 160 to 180 F, followed immediately by a 10-sec dip in the second of the above baths, operated at 120 to 140 F. Failure to preheat the castings results in no coating in 10 sec.

Following immersion the parts shall be removed, allowed to drain for not less than 5 sec nor more than 30 sec, then washed thoroughly in cold running water, followed by a dip in hot water at 160 to 180 F to facilitate drying. The parts shall not be allowed to drain following the chromate treatment for more than the specified 30 sec; excessive drainage times result in powdery coatings of poor value as paint bases. Such coatings also result from the use of hot-water rinses, the temperatures of which are in excess of 180 F. The parts shall preferably be painted immediately after they are dry. The solution may be held in type 316 stainless steel, or ceramic tanks or in steel tanks lined with synthetic rubber or vinyl-base materials. Tanks of ASTM alloy No. 990A, or its Aluminum Association equivalent, Alloy No. 1100 aluminum are satisfactory for the non-fluoride-containing pickle.

(*c*) *Revivification of Solutions.*—Sluggish reaction with the metal, associated with pale yellow, lustrous coatings, indicates that the solution is depleted. Revivification is accomplished by the addition of dichromate and nitric acid, to raise the dichromate again to 1.5 lb per gal and the free nitric acid to levels indicated in the table below. Revivification shall be made when the free nitric acid content of the solution is depleted to 0.5 pt per gal (see Section 11 for analytical procedure) and shall take place once only for M1 and ZK60A alloys,[4] and not more than six times for other alloys if good paint-base properties are desired. Excessive use of the solution or too many revivifications result in smooth, lustrous coatings not possessing the degree of etch necessary for the best paint adhesion with conventional primers. Revivify in accordance with the following table:

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[5] Conforming to Class I, Type I treatments are the Dow No. 1 process, the AMC "A" process, and the Type I process of Military Specification MIL-M-3171A.

Revivification Number	Adjust Concentration of HNO_3 to:
1	1.3 pt per gal
2	1.1 pt per gal
3 to 6	0.9 pt per gal

Class I, Type II (Sealed Chrome Pickle)[6]

4. (*a*) *Scope.*—Class I, Type II treatment is applicable to all types and forms of magnesium-base alloys, subject only to the limitations of the Class I, Type I treatment, since it is essentially the same process as the latter, followed by sealing. In protective qualities and as a paint base, this treatment is somewhat superior to that of Class I, Type I treatment under severe exposure conditions (Note 5).

(*b*) *Procedure.*—Following chrome pickling as specified under Section 3 and rinsing in cold water, the parts shall immediately be transferred to a boiling solution conforming to the following composition:

Sodium dichromate ($Na_2Cr_2O_7 \cdot 2H_2O$)	1.5 lb
Calcium or magnesium fluoride (CaF_2 or MgF_2)	⅓ oz
Water (Note 4)	to 1 gal

The parts shall be boiled in the above solution for 30 min, after which they shall be rinsed in cold running water, followed by a rinse in hot water at a temperature of not less than 160 F nor more than 180 F, to facilitate drying. Preferably the paint coating shall be applied immediately after the parts are dry. The solution may be held in a steel tank.

NOTE 5. *Causes of Defective Coatings.*—The following information is intended to provide guidance on the causes of the most usual defects arising in the application of either Class I, Type I or Class I, Type II coatings:

(*a*) *Spotted Coatings* are caused by ineffective preliminary degreasing or by the presence of excessive surface contamination not removed prior to or during the chrome pickling treatment, or both.

(*b*) *Nonadherent Powdery Coatings* are caused by:

(*1*) Too long an interval between removal from the chrome pickle and rinsing,

(*2*) Ratio of acid to sodium dichromate too high,

(*3*) Temperature of the solution or of the hot-water rinse too high,

(*4*) Metal improperly degreased, or

(*5*) Solution revivified too many times.

Class I, Type III[7]

5. (*a*) *Scope.*—Class I, Type III treatment is applicable to all types and forms of magnesium-base alloys except M1 alloy[4] and certain rare-earth alloys similar to EK30A.[4] It produces in itself no appreciable dimensional change and is, therefore, applicable to parts machined to fine tolerances. The treatment is applicable to magnesium containing inserts of, or attached to, other metals, but in such cases the preliminary treatment (Paragraph (*b*)) shall be in the bifluoride bath (Solution No. 2) described in Paragraph (*b*), Item (*2*) (Note 6).

NOTE 6: *Causes of Defective Coatings.*—The following information is intended to provide guidance on the causes of the more usual defects arising in the application of coatings from the Class I, Type III treatment.

(*a*) *Nonadherent Powdery Coatings* are caused by:

(*1*) Over-dilution of the hydrofluoric acid or acid fluoride solution,

(*2*) Low pH (less than 4.0) of the dichromate solution,

(*3*) Insufficient precleaning of the metal surface, and

(*4*) Direct contact between the steel tank containing the dichromate solution and the article being treated.

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[6] Conforming to Class I, Type II treatments are the AMC "L" process, the Dow No. 10 process, and Type II treatment of Military Specification MIL-M-3171A.

[7] Conforming to Class I, Type III treatments are the AMC "G" process, the Dow No. 7 process, and the Type III treatment of Military Specification MIL-M-3171A.

(*b*) ***Failure to Coat and Nonuniform Coatings*** are caused by:

(*1*) High pH of the dichromate solution,

(*2*) Low concentration of the dichromate,

(*3*) Insufficient precleaning of the metal surface,

(*4*) Omission of fluoride treatment,

(*5*) Use of an unsuitable alloy for the treatment, for instance, M1 alloy,

(*6*) Excessive immersion time in the hydrofluoric acid solution or use of an H_2F_2 concentration in the dichromate solution in excess of 0.2 per cent,

(*7*) Insufficient rinsing after the hydrofluoric acid dip, and

(*8*) Insufficient heating of the dichromate solution (minimum temperature should be 200 F).

(*b*) *Procedure.*—Following cleaning as prescribed in Section 2, the parts shall be treated first by immersion at 70 to 90 F in one or other of the following solutions:

(*1*) *Solution No. 1:*

Hydrofluoric acid (60 per cent H_2F_2)	24 fl oz
Water (Note 4)	to 1 gal

(*2*) *Solution No. 2:*

Sodium, potassium, or ammonium acid fluoride ($NaHF_2$, KHF_2, or NH_4HF_2)	6⅔ oz
Water (Note 4)	to 1 gal

Solutions No. 1 and No. 2 may be held in tanks consisting of steel lined with lead or rubber.

In solution No. 1, AZ31A and AZ31B alloy parts[4] shall be immersed for from 30 sec to 1 min; all other alloys shall be immersed for 5 min. In solution No. 2, all the alloys shall be immersed for 5 min. Solution No. 2 is suitable for use with all forms of magnesium alloys except those castings which have *not* been acid-pickled after blasting; these castings shall be treated in the hydrofluoric acid solution (solution No. 1).

Following one or other of the above treatments, the parts shall be rinsed thoroughly in cold running water and transferred to a solution conforming to the following composition:

Sodium dichromate ($Na_2Cr_2O_7 \cdot 2H_2O$)	1 to 1.5 lb
Calcium or magnesium fluoride (CaF_2 or MgF_2)	⅓ oz
Water (Note 4)	to 1 gal

The above solution shall be operated at boiling-point and the parts shall be immersed therein for 30 min, following which they shall be thoroughly rinsed in cold running water, followed by a hot-water dip to facilitate drying. Preferably, the paint coating shall be applied as soon as possible thereafter. A mild-steel tank is suitable for holding the above solution.

(*c*) *Maintenance of Solutions:*

(*1*) *Fluoride Solutions.*—The concentration of free hydrofluoric acid in the solutions of Paragraph (*b*), Items (*1*) and (*2*) shall be kept constant by the addition, as required, of either hydrofluoric acid or acid fluoride, respectively. See Section 9 for the analytical procedure.

(*2*) *Dichromate Solution.*—The pH of the dichromate solution shall be kept within the limits 4.0 to 5.5, or 4.0 to 4.8 in the case of AZ31A or AZ31B alloy,[4] by addition of chromic acid as required. The level shall be maintained by the addition of water. The solution shall be maintained saturated, with respect to either magnesium or calcium fluoride, by continuous immersion of a cloth bag containing excess of one or other of these compounds.

SURFACE PREPARATION PROCEDURES—ANODIC

Class II, Type I (Galvanic Dichromate Treatment)[8]

6. (*a*) *Scope.*—This treatment pro-

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[8] Conforming to Class II, Type I treatment are the AMC "K" process, the Dow No. 9 process, and Type IV treatment of Military Specification MIL-M-3171A.

duces black coatings of good protective and paint-base qualities, and is applicable to all alloys and forms of magnesium, including M1 alloy,[4] with or without attachments of other metals. No appreciable dimensional change results, and hence the treatment is also applicable to articles machined to fine tolerances.

(*b*) *Procedure.*—Following cleaning as prescribed in Section 2, the articles shall be treated in fluoride solution No. 1 or No. 2 as prescribed in Section 5(*b*). The latter solution shall be used when other metals are attached to the magnesium. After rinsing, the articles shall be immersed in a solution conforming to the following composition:

Ammonium sulfate ($(NH_4)_2SO_4$)	4 oz
Sodium dichromate ($Na_2Cr_2O_7 \cdot 2H_2O$)	4 oz
Ammonia (NH_4OH) (sp gr 0.880)	⅓ fl oz
Water (Note 4)	1 gal

The above solution shall be operated at 120 to 140 F. The articles shall be made the anodes in the solution, with the tank, if of mild steel, acting as cathode, or with separate steel cathodes if the tank is lined with nonmetallic materials. No separate generator is necessary, but the magnesium parts must be electrically connected with the tank, or with the separate cathode plates through an external connection, care being taken that the parts do not make direct contact with the cathode material.

The time of treatment shall be such that a uniform black coating is obtained on the articles. This takes from 10 to 30 min, and the consumption of from 70 to 150 amp-min per sq ft. An anodic current density of not more than 10 amp per sq ft is desirable.

Following the treatment the parts shall be rinsed thoroughly in cold running water, followed by a hot-water dip to facilitate drying.

(*c*) *Maintenance of Solutions:*

(*1*) *Fluoride Solutions.*—The fluoride solutions shall be maintained as under Section 5(*c*), Item (*1*). See Section 9 for the analytical procedure.

(*2*) *Sulfate - Dichromate Solution.*—The pH of this solution shall be maintained between 5.6 and 6.2 by periodic additions of a solution containing 5 per cent by weight of both chromic anhydride (CrO_3) and concentrated sulfuric acid (H_2SO_4, sp gr 1.84).

Class II, Type II[9]

7. (*a*) *Scope.*—This treatment is applicable to all forms and alloys of magnesium. Many aluminum alloys will anodically polarize in the treatment solution, and hence magnesium parts with aluminum attachments or inserts can be anodized. With aluminum alloys containing copper, however, the coating of the magnesium will proceed only if the area of the aluminum alloy is small compared to that of the magnesium. Metals other than aluminum or its base alloys shall not be in contact with the magnesium.

(*b*) *Coating Properties.*—The anodic coating consists of two phases: the first-formed phase is greenish-tan in color and about 0.0002 in. thick; whereas the second phase, formed only at higher voltages following the production of the tan coating, is fused and vitreous in nature, dark-green in color, and of thickness about 0.0012 in., causing a dimensional increase of about 0.001 in. per surface. The corrosion-resistance and paint-base characteristics are similar, and excellent for both the above phases. The thicker coating possesses high abrasion resistance, but spalls under compression deformation. The dark-green coating can be partially impregnated with low-viscosity, penetrating, organic coating materials, a procedure that considerably reduces its spalling tendency (Note 7).

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[9] Conforming to Class II, Type II treatment is the Dow No. 17 process.

NOTE 7.—The dark-green coating of Class II, Type II treatments is recommended in preference to the thin tan coating only when:

(*a*) Preliminary removal of surface contamination is not convenient.

(*b*) The highest degree of abrasion-resistance is required from the coating.

(*c*) A dimensional increase (see above) can be tolerated.

(*d*) The article will not be subjected in service to impact, deformation, or flexing likely to cause spalling of the coating.

(*c*) *Procedure.*—The thin tan coating is formed with a current consumption and in a time approximately one quarter of that required to form the dark-green coating. The latter forms only after the voltage across the solution has reached the spark potential. During the ensuing coating formation, surface contamination, including graphite, is concurrently removed. Thus, articles intended to receive the dark-green coating need not first be cleaned. On the other hand, the formation of the thinner tan coating does not concurrently remove surface contamination. Since such contamination can seriously reduce corrosion resistance, it is mandatory that a preliminary cleaning, including acid pickling (Section 2(*b*)) shall be given to articles intended to receive the lower-voltage tan coating.

Parts shall be racked, cleaned as appropriate (see above), and anodized either by direct or alternating current in an electrolyte conforming to one of the following compositions, respectively:

	Direct Current	Alternating Current
Ammonium acid fluoride (NH_4HF_2) . . .	32 oz	40 oz
Sodium dichromate ($Na_2Cr_2O_7 \cdot 2H_2O$) . .	13.3 oz	13.3 oz
Phosphoric acid (85 per cent H_3PO_4) . . .	11.5 fl oz	11.5 fl oz
Water (Note 4)	to 1 gal	to 1 gal

The temperature of the solution shall preferably be from 160 F to 180 F. It will not operate below 140 F, but temperatures higher than 180 F do not deleteriously affect the results. After anodizing, the parts shall be rinsed in running cold water, followed by a short immersion in hot water, or by treatment with hot air, to facilitate drying. Preferably, the paint coating shall be applied immediately after the parts are dry.

(*d*) *Power Requirements.*—A current consumption of from 50 to 500 amp-min per sq ft is necessary, according to both the composition of the alloy being treated and the type of coating desired. Thus, in the case of the dark-green coating on AZ31 alloy, for a 10-min treatment with alternating current, a current density of 30 amp per sq ft is applied; but greater or lesser currents may be utilized by varying the time of treatment as, for example, application of 50 amp per sq ft for 6 min. On the other hand, for the same alloy, the thin tan coating may be applied with alternating current, with a current consumption of 75 amp-min per sq ft, equivalent to a treatment time of only $1\frac{1}{2}$ min at 50 amp per sq ft. As the coating forms, the resistance of the circuit increases, and consequently the voltage must be increased to maintain a constant current density. Normally the voltage across the bath will reach from 75 to 95 for the dark-green coating (according to alloy composition) and from 60 to 75 for the thin tan coating. In carrying out the treatment, a constant current control is a considerable advantage, inasmuch as the total treatment time can be preset and manual control of the voltage and current then become unnecessary.

As guides to the current consumptions with various alloys, some data are presented in Table I. It will be appreciated that rigid current consumptions in the formation of the thin tan coating cannot be specified, since this coating can be varied in thickness considerably between say 0.0001 in. and 0.0005 in., and still be formed below the critical spark potential at which the dark-green vitreous coating begins to develop.

(*e*) *Solution Control.*—With depletion of the bath upon use, the final voltage required to impress the above currents on the article will slowly rise, but even for the full green coating at least 20 sq ft of surface can be treated per gallon of solution before any change in the final voltage is experienced; and up to 40 sq ft per gal can be treated before revivification of the solution becomes necessary.

TABLE I.—CURRENT CONSUMPTION WITH VARIOUS ALLOYS.

Alloy	Alternating Current				Direct Current			
	Voltage		Amp-min per sq ft		Voltage		Amp-min per sq ft	
	Dark-Green	Thin Tan	Dark-Green	Thin Tan	Dark-Green	Thin Tan	Dark-Green	Thin Tan
AZ31A or B........	90	70	300	75	95	75	190	50
AZ61A.............	90	68	400	...	95	72	300	...
AZ63A.............	80	68	600	100	84	72	400	70
AZ91A.............	75	64	480	...	80	69	280	...
AZ92A.............	75	64	600	...	80	69	400	...
EK30A.............	90	70	400	...	95	75	300	...
ZK60A.............	85	70	300	...	90	75	190	...

Prior to revivification, analyses shall be made for dichromate and phosphoric acid (see Sections 10 and 12), and differences so shown from the original composition shall be made up by additions of sodium dichromate and phosphoric acid.

A simple method for the determination of fluoride in the solution is not yet available. The fluoride concentration, however, is not critical, provided it always exceeds minimum concentration, below which pitting of the articles may ensue during their treatment. When revivifications are made by additions of sodium dichromate and phosphoric acid, a concurrent addition shall be made of ammonium acid fluoride, equal to three times the weight of sodium dichromate added. Secondly, if pitting of the articles should occur under treatment, it is an indication that the fluoride content of the solution is too low, and about 5 oz per gal of ammonium acid fluoride shall then be added in order to relieve the trouble.

(*f*) *Tank and Rack Materials.*—Mild steel is suitable for tanks and heating coils. Other metals, such as copper, aluminum, lead, zinc, monel, and stainless steels are attacked by the electrolyte. When direct current is used the tank itself can be the cathode, but, when alternating current is used, at least two magnesium parts are necessary, of area ratios not more than 3 to 1, each one serving as electrode. The articles shall not contact the tank, otherwise pitting will ensue.

Racks shall be normally made of magnesium alloy, but aluminum alloys containing magnesium, such as ASTM alloy No. GR20A, or its Aluminum Association equivalent, alloy No. 5052, can be used if desired, provided they are not allowed to be in the solution on open circuit. A spring clip with magnesium ends shall be used for providing contact with thin sheet material, the liquid level not being allowed to extend beyond the limit of the magnesium ends of the clip.

No solution-line attack is experienced on magnesium. Magnesium racks may be stripped by immersion for 2 min in hot 20 per cent chromic acid solution or by immersion at room temperature for about 8 min in the chromic acid-nitrate solution bath described in Section 2 (*b*), Item (*3*).

Class II, Type III[10]

8. (*a*) *Scope.*—This treatment is applicable to all forms and alloys of magnesium, free from attachments or inserts of other metals.

(*b*) *Coating Properties.*—The anodic coating consists of two phases: The first-formed subcoating phase is a light tan in color, and causes a dimensional increase of about 0.0002 in. per side; the second and thicker phase, formed at higher voltages, is dark brown and causes a dimensional increase of from 0.001 in. to 0.0012 in. per surface. The paint-base characteristics are similar, and excellent for both the above phases.

The dark-brown coating is hard and highly abrasion-resistant, but it spalls under compression deformation and its formation can cause a loss of fatigue strength of the metal. Such loss is negligible with metal of thickness 0.1 in. or over, rising to 3 per cent and 9 per cent for thicknesses of 0.094 in. and 0.040 in., respectively, all under reversed bending and corrected for coating thickness. No loss of fatigue strength arises from application of the thin tan coating, and neither is this coating subject to spalling.

(*c*) *Procedure.*—Parts shall be racked and cleaned with hot alkaline cleaner (*Section 2* (*a*)) or chromic acid (Section 2 (*b*), Item (1)), or both, as appropriate, and anodized with alternating current in an electrolyte conforming to the following composition:

Potassium hydroxide (KOH)	18 to 20 oz
Aluminum hydroxide ($Al(OH)_3$) soluble in hot KOH solution (Note 8)	4 oz
Potassium fluoride (K_2F_2)	4.5 oz
Trisodium phosphate (Na_3PO_4)	4.5 oz
Potassium manganate (K_2MnO_4) (Note 9)	2.5 oz
Water (Note 4)	to 1 gal

The constituents of the electrolyte shall be dissolved in the above order. The temperature of the electrolyte shall be maintained between the range 77 to 100 F.

NOTE 8.—If an exceptionally hard, abrasion-resistant dark-brown coating is desired, the aluminum hydroxide in the above electrolyte shall be increased to 6 to 7 oz per gal.

NOTE 9.—Potassium manganate (K_2MnO_4) in the above electrolyte may be replaced with an equal weight of potassium permanganate ($KMnO_4$), but in such case the permanganate shall be completely dissolved in water before it is added to the solution and an additional 1½ oz of potassium hydroxide shall also be added. During operation of the bath, the permanganate is slowly reduced to manganate, complete conversion taking place after a metal area of approximately 2 sq ft per gal of electrolyte has been treated.

(*d*) *Post-Treatment.*—After the electrolytic treatment, the parts shall be well rinsed in water and immersed for 1 min at room temperature in a solution consisting of 2.7 oz of sodium dichromate ($Na_2Cr_2O_7 \cdot 2H_2O$) and 13.3 oz of ammonium bifluoride (NH_4HF_2) per gallon of solution. Following the immersion, the parts shall be removed and dried without rinsing. Painting shall follow at any convenient time after the parts have dried.

(*e*) *Power Requirements.*—The practical range of current density is from 15 to 20 amp per sq ft, with voltages rising to from 70 to 86 (depending on alloy) for the hard brown coating and to 58 v for the thin tan coating. In this current density range, the terminating voltage is reached in from 45 to 60 min for the hard brown coating and in 10 min or less for the thin tan coating. The voltage shall be raised quickly from zero to 40 or more, and the current then adjusted and retained at the density desired. Normally the voltage will rise to 55 or more within 1 min, but as the coating forms, the resistance of the circuit increases and the voltage must be raised continuously to maintain the desired current density (Note 10).

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[10] Conforming to Class II, Type III treatment is the HAE process.

NOTE 10.—If, at low initial voltages, the current remains high and coating formation does not proceed, a "surge" current shall be imposed by rapidly raising the voltage to 40, followed by immediately lowering it to zero. The voltage shall then be adjusted in the normal manner to maintain the desired current density.

(*f*) *Solution Control.*—The operating life of the electrolyte is extremely long, and under normal conditions of use it need never be discarded. Manganate and aluminate deplete slowly with use, whereas the depletion of fluoride and phosphate is extremely slow.

In the case of the dark-brown coating, a lightening of the normal color indicates a depletion of manganate in the solution, and additions shall then be made of 1 oz of potassium manganate plus $1\frac{1}{2}$ oz of aluminum hydroxide for each gallon of solution. The aluminum hydroxide shall be added after first dissolving it in a solution containing a weight of potassium hydroxide equal to that of the aluminum hydroxide to be added.

A rough coating indicates either a low, or an excessively-high, alkalinity in the electrolyte. The alkalinity shall be maintained between 10 and 12 per cent free KOH, as determined by the method described in Section 13.

(*g*) *Tank and Rack Materials.*—Plain black iron is suitable for tanks and cooling coils. Suspension of the parts shall be by magnesium-base alloy clips or the like and at the solution level these shall be protected by electroplaters' vinyl tape. Magnesium racks may be stripped of coating when necessary by immersion in hot 20 per cent chromic acid solution (Note 11).

Parts to be treated shall be divided into two batches each of approximately the same surface area. Each of the batches shall be racked and connected electrically to one or other of the inlet terminals of the power supply so that the magnesium parts constitute both electrodes of the electrolysis bath (Note 12).

NOTE 11: *Safety Precautions.*—Consideration should be given to the health hazards associated with the procedures covered by this specification. Precautions must be taken to avoid skin contact with any of the solutions involved, and to avoid inhalation of vapors, fumes, or spray arising therefrom. Baths containing dichromate or fluoride shall be fitted with exhausts to remove all spray arising therefrom.

NOTE 12: *Repair Treatments.*—Pretreatment films that have been damaged shall be repaired before painting. Repair shall be effected by one of the treatments below. The phosphate touch-up solution of Paragraph (*b*) below necessitates special preparation, whereas a chrome pickle solution is often already available. Nevertheless, for brush-on treatment, the phosphate solution possesses the advantages of being nontoxic and nonirritant.

(*a*) *Chrome Pickle Repair Treatment.*—The chrome pickle solution for Class I, Type I treatments (for wrought parts) shall be copiously and continuously applied to the damaged area by brush for at least 1 min. The treated surface shall then be thoroughly flushed with water and, when dry, rubbed vigorously with a clean dry rag to remove loose powdery matter which, if allowed to remain, would deleteriously affect the adhesion of superimposed paint.

(*b*) *Phosphate Repair Treatment.*—A solution shall be used conforming to the following composition:

Monoammonium phosphate ($NH_4H_2PO_4$)	16 oz
Ammonium sulfite ($(NH_4)_2SO_3 \cdot H_2O$)	4 oz
Butyl cellosolve	13 to 20 fl oz
Water (see Note 4)	1 gal

The above solution shall be applied copiously to the damaged area by brush in sufficient quantity to keep the surface wet for at least 1 min, or until a medium to dark-gray continuous coating is formed. The treated metal surface shall then be well flushed with water and allowed to dry before the paint is applied

ANALYTICAL PROCEDURES

Fluoride Solutions

9. This method is applicable to the relevant baths of Class I, Type III (Section 5) and Class II, Type I (Section 6) treatments. The concentrations of both hydrofluoric acid (HF) and bifluoride shall be determined by titration with 1 *N* sodium hydroxide (NaOH) solution, using phenolphthalein as indicator.

(*a*) Maintain the hydrofluoric acid solution described in Section 5 (*b*) by additions of fresh acid to give a titration of between 10 and 20 ml of 1 *N* NaOH solution per 2 ml of sample. Maintain the bifluoride solution described in Section 5 (*b*) so that 10 ml thereof gives a titration of 4.5 to 5.5 ml of 1 *N* NaOH solution.

(*b*) For the hydrofluoric acid the sample shall be drawn into a wax-lined pipet, discharged therefrom into at least 100 ml of distilled water, and titrated immediately. A rubber bulb, or a length of rubber tubing fitted to the suction end of the pipet, shall be used in drawing the sample into the pipet.

Dichromate Solutions

10. This method is applicable to solutions of the following treatments: Class I, Types I, II, and III; and Class II, Types I and II (Sections 3, 4, and 5; and Sections 6 and 7, respectively).

(*a*) Add 1 ml of the dichromate solution concerned to 150 ml of distilled water, and mix therewith 5 ml of concentrated hydrochloric acid (HCl, sp gr 1.19) and 5 g of potassium iodide (KI). After at least 2 min, titrate the liberated iodine in the solution with 0.1 *N* sodium thiosulfate ($Na_2S_2O_3$) solution using starch as an internal indicator.

(*b*) *Calculation:*

$$A \times 0.0415 = B$$

where:

A = milliliters of 0.1 *N* $Na_2S_2O_3$ solution, and

B = pounds per gallon of sodium dichromate ($Na_2Cr_2O_7 \cdot 2H_2O$).

Nitric Acid Solution

11. This method is applicable to Class I, Types I and II treatment solutions.

(*a*) Mix 50 ml of distilled water with 1 ml of the solution in question, and titrate the mixture with 0.1 *N* sodium hydroxide (NaOH) solution to a pH of 4.0 to 4.05, using a pH meter with a glass electrode.

(*b*) *Calculation:*

$$A \times 0.0505 = B$$

where:

A = milliliters of 0.1 *N* NaOH solution, and

B = pints per gallon of nitric acid (HNO_3).

Phosphoric Acid Solution

12. This method is applicable to the Class II, Type II treatment solution. The phosphoric acid is precipitated as ammonium phosphomolybdate, which is dissolved in an excess of sodium hydroxide, such excess being titrated with standard acid. Interference from fluoride is prevented by converting it to fluoborate, by addition of boric acid.

(*a*) *Molybdate Reagent.*—Prepared as follows: mix 118 g of 85 per cent molybdic acid (H_2MoO_4) with 400 ml of distilled water, add 80 ml of ammonium hydroxide (NH_4OH) solution (sp gr 0.880) with stirring, and filter when solution is complete. Slowly pour the filtrate, with stirring, into a cold mixture of 400 ml of concentrated nitric acid (HNO_3, sp gr 1.42) and 600 ml of water.

Allow the solution to stand over night, and filter.

(*b*) *Procedure.*—Dilute a 14-ml weighed sample of the solution under examination to 500 ml, and then further dilute 50 ml of this diluted solution to 500 ml. Then again dilute 25 ml of the latter solution to 100 ml, and add 10 g of ammonium nitrate (NH_4NO_3), 1 g of boric acid (H_3BO_3), and 15 ml of concentrated nitric acid (HNO_3, sp gr 1.42). Heat the mixture to 104 F and slowly add 40 ml of molybdate reagent. Allow the resulting precipitate of ammonium phosphomolybdate to stand for 30 min, and then filter through a fine-texture, low-ash paper.[11] Wash the precipitate free from acid by means of a 1 per cent solution of potassium nitrate (KNO_3), and mix, together with the filter paper, with an excess of 0.1 *N* sodium hydroxide (NaOH) solution, using phenolphthalein as indicator. Titrate the excess of NaOH with 0.1 *N* hydrochloric acid (HCl).

(*c*) *Calculation:*

Phosphoric acid (H_3PO_4), per cent =

$$\frac{A - B}{C} \times 0.426$$

where:

A = milliliters of 0.1 *N* NaOH solution,

B = milliliters of 0.1 *N* HCl, and

C = grams of sample in aliquot.

Free Alkali

13. This method is applicable to the Class II, Type III solution. Manganate, fluoride, and phosphate are precipitated by barium nitrate addition, and the filtrate therefrom is titrated with standard acid.

(*a*) Pipet a 2-ml sample of the solution under examination into a 25-ml glass-stoppered graduated cylinder, followed by addition of 10 ml of 0.25 *N* sodium hydroxide (NaOH) solution and 0.5 g of barium nitrate ($Ba(NO_3)_2$) crystals. Shake this mixture vigorously for 1 min, filter through a medium-texture, low-ash paper,[12] and wash the precipitate free from alkali, preserving all filtrate. Using a pH meter with a glass electrode, titrate the free alkali in this filtrate to pH 10.5, with 0.25 *N* hydrochloric acid (HCl).

(*b*) *Calculation:*

Potassium hydroxide, per cent =

$$2.805 \times \left(\frac{A}{4} - 2{\cdot}5\right)$$

where:

A = milliliters of acid used.

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[11] Whatman No. 42, or equivalent, paper has been found satisfactory for this purpose.

[12] Whatman No. 40, or equivalent, paper has been found satisfactory for this purpose.

APPENDIX III

SUMMARY OF COOPERATIVE DATA ON STUDY OF METHOD OF TEST FOR FLASH POINT OF VOLATILE FLAMMABLE MATERIALS BY TAG OPEN CUP APPARATUS (D 1310 - 55 T)[1]

JULY, 1954, TO FEBRUARY, 1956

Data reported in February, 1955, showed differences between laboratories sometimes as large as 10 F which were difficult to explain. These corrected data are shown in Table I. The magnitude of the correction in column *6* shows the degree of difficulty as it appeared in February, 1955. A program of interlaboratory visitation was planned to find out whether apparatus, operator, or location (drafts, etc.) was causing the difficulty. The data given in Table II show that apparatus is the important variable.

Since the height of the taper above the cup seemed important, one laboratory equipped a tester with a micrometer adjustment for the level of the swing arm. Using this equipment, the data in Table III were obtained which show that increasing the height of the taper by 0.01 in. can increase the flash by 1.3 to 2.0 F.

The evidence in Tables II and III made it unlikely that it would be practical to seek improvement in apparatus by closer manufacturing tolerances.

TABLE I.—FLASH POINT DATA BY REVISED METHOD D 1310.[a]
Reported February to March, 1955.

Laboratory	*p*-Xylene		Isopropyl Alcohol		Butyl Acetate		Ethyl Acetate		Correction Used	Spread of Standards
Celanese	92	+1	71	−1	91	+1	24	−4	−6	21
Hercules	91	0	73	+1	88	−2	23	−5	−3	18
Shell—Houston	91	0	73	+1	91	+1	31	+3	−5	18
Shell—Denver	92	+1	70	−1	92	+2	28	+1	+5	22
Southern RR	92	+1	71	−1	92	+2	27	−1	+3	21
Sun Oil Co.	91	+0	71	0	91	+1	27	0	+3	20
Bakelite	89[b]	−2	75[b]	+3	88	−2	35	+7	−1	14[b]
Carbide and Carbon[c]										
Bur. of Explosives	92	+1	70	−2	89	−1	..	..	−0.5	22
Average	91		72		90		28		3	20
Maximum	92		75		93		35		+5	
Minimum	89		70		88		23		−6	
Spread	4		5		5		14		11	
Standard Deviation		1.1		1.6		1.7		4.1	...	

[a] Revised procedure based on *p*-xylene = 92 F flash, isopropyl alcohol = 71 F flash, and average correction term.

[b] Spread of 14 between *p*-xylene and isopropyl alcohol is less limit of 15 suggested in proposed revision. Omitted in calculating average difference between standards.

[c] Data omitted since the difference between flash of *p*-xylene and isopropyl was only 6 F.

TABLE II.—COMPARISON OF APPARATUS, OPERATORS, AND LOCATIONS.

	p-Xylene					Isopropyl Alcohol		*n*-Butyl Acetate	
	Carbide Operator	Sun Oil Operator	Hercules Operator	Bakelite Operator	Bur. of Explosives Operator	Bur. of Explosives Operator	Sun Oil Operator	Bur. of Explosives Operator	Sun Oil Operator
Carbide Tester, Swing Arm Type	100[a] 98[a] 96[c] 96[c] 100[e] 98[i]	97[a] 97[a] 98[c]	98[c]	98[e]	100[i]				
Sun Oil Tester, Swing Arm Type	94[a] 92[bc]	90[a] 93[a] 94[ab] 94[bc] 93[bc] 93[bcd] 92[k] 92[k]	92[bc]				70[k] 70[k]		90[k] 88[k]
Hercules Tester, Swing Arm Type	100[bc]	100[bc]	98[c] 100[bc]						
Bakelite Tester, Swing Arm Type	106[eg] 92[eg] 96[e]			99[ef] 98[ef] 97[ebh] 94[ebh]					
Bur. of Explosives Tester (Old Model, no swing arm)					96[i] 92[i] 96[k] 90[k]	68[k] 72[k]		90[k] 92[k]	

[a] Determined at Marcus Hook.

[b] Carbide leveling device used and taper adjusted to same level as that on Carbide tester.

[c] Determined at Wilmington.

[d] Sun Oil tester except that glass cup from Carbide tester was used.

[e] Determined at Bound Brook.

[f] Determined in the same manner and with the equipment in the same condition normally used at Bakelite.

[g] During these determinations, the operator was not sure that the glass cup was level.

[h] Technique used conformed more nearly to procedure as written.

[i] Determined at Bureau of Explosives Lab., South Amboy, first visit.

[k] Determined at Bureau of Explosives Lab., South Amboy, second visit.

Calibration with standard liquids seemed a good way of canceling out apparatus variables and also the effect of altitude. Flash points as determined at sea level (Bureau of Explosives Laboratory, South Amboy, N. J.) have been accepted for many years as controlling in the classification for interstate shipment of flammable or non-flammable liquids.

p-Xylene and isopropyl alcohol were chosen as standards since one is about 10 F higher and one about 10 F lower than the 80 F dividing line between flammable and non-flammable liquids.

Based on the cooperative work, *p*-xylene has been assigned a value of 92 F flash and isopropyl alcohol 71 F flash; average 81.5 F. The average value shown by the Bureau of Explosives Laboratory on these two compounds (Table II) is 81.7 F. The new standards adhere to the level of results established by the Bureau of Explosives.

The data given in Tables I and IV have been corrected on the basis of the standard values for *p*-xylene and isopropyl alcohol. For materials other than the standards flashing in the range 60 to 110 F, the standard deviation for repeatability (1956 data) is in the range 2.7 to 3.3, and the maximum spread between laboratories is not over 10 F. Below 60 F, larger deviations are shown.

Table IV also gives standard deviations for repeatability (one laboratory) which comes out in the range 0.9 to 2.9 F.

TABLE III.—DIFFERENCES IN TAPER HEIGHT ABOVE CUP.[a]

Distance of Taper Above Cup, in.	Flash Point, deg Fahr	
	p-Xylene	Butyl Acetate
0.061	80	84
0.125	92	96
0.176	96	106
0.227	102	117
0.277	102	126
Slope	1.3 F per 0.01 in.	2.0 F per 0.01 in.

[a] Data obtained by Carbide and Carbon Co., with micrometer adjustment on taper.

The improvements made in the last year bring all the cooperative data within the stated precision limits of the method. No change in the wording of the precision section is recommended this year except in the note relating to the effect of barometric pressure.

Tables I, II, III, and IV are presented in support of the recommended revisions of Method D 1310.[1]

Data support the conclusion that differences in equipment are more important than differences between operators or locations.

Data show that very small differences in taper height above cup has a large effect on flash.

Tables III and IV support the conclusion that calibrating liquids must be used to eliminate differences between equipment.

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[1] 1955 Book of ASTM Standards, Part 4, p. 425.

TABLE IV.—COOPERATIVE DATA—JULY, 1955, TO FEBRUARY, 1956.

Tag Open Cup Flash, D 1310 Revised.[a]

Laboratory	Para-xylene		Iso-propyl Alcohol		Isobutyl Alcohol		Ethyl-Benzene		Toluene		Mineral Spirits		Correction[a] Used	Spread of Standard	Standard Deviation, Repeatability	No. of Data
Bakelite	94	+2	69	−2	88	−5	83	0	54	−1	107	−4	−3	25	1.3	26
Shell—H	91	−1	72	+1	93	0	80	−3	(44)	−11	106	−5	−1	19	0.9	30
Shell—NJ	93	+1	70	−1	93	0	81	−2	54	−1	111	0	0	23	1.4	30
Shell—D	91	−1	72	+1	98	+5	84	+1	55	0	116	+5	+8	19	1.3	30
Carbide and Carbon																
A	90	−2	74	+3	89	−4	83	0	56	+1	108	−3	−4	16	0.9	42
B	92	0	72	+1	96	+3	81	−2	(70)	+15	108	−3	−5	20	1.1	52
Sun Oil	94	+2	68	−3	91	−2	81	−2	53	−2	112	+1	+2	26	1.1	29
Southern RR	90	−2	73	+2	94	+1	80	−3	61	+6	111	0	0	17	1.7	29
Celanese	92	0	72	+1	93	0	89	+6	50	−5	114	+3	−3	20	1.4	29
Hercules	93	+1	70	−1	97	+4	83	0	52	−3	113	+2	0	23	2.9	22
Average	92		71		93		83		55		111			21	1.4	32
Maximum	94		74		98		89		70		116			26	2.9	
Minimum	90		68		88		80		44		106			16	0.9	
Spread	4		6		10		9		26		10			10	2.0	
Average Deviation	1.2		1.6		2.4		1.9		4.5		2.5					
Standard Deviation of Reproducibility	1.5		1.9		3.3		2.7		6.9		3.3					

Shell H = Houston

Shell NJ = Sewaren

Shell D = Denver (old equipment)

Carbide A and B are different operators. Each operator used two test units. On toluene and isobutyl alcohol operators differ, but test units do not.

[a] Revised method uses para-xylene 92 Flash and isopropyl alcohol 71 Flash as standard and a single correction factor rounded to the nearest whole (smaller) number.

REPORT OF COMMITTEE D-2

ON

PETROLEUM PRODUCTS AND LUBRICANTS*

This report contains the recommendations of the subordinate groups of Committee D-2 on Petroleum Products and Lubricants. The recommendations have been approved by the committee, in accordance with the regulations of the Society, and are presented herewith.

PROPOSED METHODS TO BE PUBLISHED AS INFORMATION

It is recommended that the following 16 proposed methods be published as information, as appended hereto[1] or as otherwise indicated:

Appendix I. Oxidation Test for Turbine Oils (Short-Term Method).

Appendix II. Sulfur in Petroleum Products by the Rapid, High-Temperature Combustion Method.

Appendix III. Active Sulfur in Cutting Fluids.

Appendix IV. Hydrocarbon Types in Gasoline by Mass Spectrometry.

Appendix V. Reduction of Observed API Gravity to API Gravity at 60 F—High-Temperature Range.

Appendix VI. Reduction of Volume to 60 F Against API Gravity at 60 F—High-Temperature Range.

Appendix VII. Density and Specific Gravity of Viscous Materials and Melted Solids by Bingham Pycnometer Method.

Appendix VIII. Density and Specific Gravity of Hydrocarbon Liquids by Lipkin Bicapillary Viscous Oil Pycnometer.

Appendix IX. Low-Temperature Torque of Ball Bearing Greases.

Appendix X. Functional Life of Ball Bearing Greases .(The method is essentially the same as Appendix I to the 1953 Report of Committee D-2, with minor equipment changes.)

Appendix XI. Reduction of Volume to 60 F Against API Gravity at 60 F—Low-Temperature Range.

Appendix XII. Reduction of Volume to 60 F Against API Gravity at 60 F—Low-Temperature Range (Abridged Table).

Appendix XIII. Measuring the Color of Petroleum Products.

Appendix XIV. Thermal Stability of Fuel Oils.

Appendix XV. Proposed Revisions of Tentative Method of Gaging Petroleum and Petroleum Products (D 1085 - 56 T).

Appendix XVI. Proposed Antiknock Ratings Over 100 Octane Number by the Research and Motor Methods.

NEW TENTATIVES

The committee recommends that the following ten methods be accepted for publication as tentative, as appended hereto[2] or as otherwise indicated:

Tentative Methods of Test for:

Emulsion Characteristics of Steam-Turbine Oils. (A similar method was published as Appendix IV to the 1954 Report of Committee D-2.)

Estimation of Heat of Combustion of Liquid Petroleum Products. (See Appendix VI to the 1955 Report of Committee D-2.)

Effect of Copper on Oxidation Rate of Grease.

Cone Penetration of Lubricating Grease Using One-Quarter Scale Cone Equipment.

Estimation of Deleterious Particles in Lubricating Grease (See Appendix IX to the 1955 Report of Committee D-2.)

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* Presented at the Fifty-ninth Annual Meeting of the Society June 17-22, 1956.

[1] These methods appear in the 1956 Compilation of ASTM Standards on Petroleum Products and Lubricants.

[2] The new tentatives appear in the 1956 Supplement to Book of ASTM Standards, Part 5.

Tentative Methods for:

Liquid Calibration of Tanks.[2a]
Calibrating Barge Tanks.[2a]
Calibrating Stationary Horizontal Tanks.[2a]
Calibrating Spherical and Spheroidal Tanks.[2a]
Calibrating Tank Car Tanks.[2a]

REVISIONS OF TENTATIVES

The committee recommends that the following 14 tentatives be revised as indicated and continued as tentative:

D 94 – 55 T,[3] Test for Saponification Number of Petroleum Products by Color-Indicator Titration:

Section 6.—Change the first sentence to read as follows:

> Blank determinations shall be made in duplicate concurrently with each set (one or more) of samples as follows: Measure accurately from a buret into the Erlenmeyer flask 25 ± 0.03 ml of alcoholic KOH and add 25 ± 1 ml of methylethylketone.

Replace the last sentence of Section 6 with the following:

> When the indicator color is discharged, add 3 drops of indicator, and if this addition of indicator restores the color, continue the titration, making further 3-drop additions of indicator, if necessary, until the end point is reached. The end point is reached when the indicator color is discharged and does not immediately reappear upon the addition of 3 drops of indicator.

Section 8.—Replace the last sentence with the above proposed new sentences for Section 6.

D 381 – 54 T,[3] Test for Existent Gum in Fuels by Jet Evaporation:

Appendix: Sections A1(a), A2, A4(a), and *A4(c).*—Change "*n*-pentane" to "*n*-heptane."

Appendix: Section A3.—Change to read: "A3. *n-Heptane,* ASTM knock test grade, conforming to the requirements given in Table I of ASTM Method D 611."

D 439 – 55 T,[3] Specifications for Gasoline:[3a]

Table I.—In *Footnote a* delete the word "automatic."

Appendix: Octane Number.—Replace the first two sentences of the final paragraph with the following:

> At the present time there is no completely satisfactory way of translating Motor and Research octane numbers into terms of a rating for all vehicles on the road. Correlation of laboratory ratings with road ratings have been developed, but at best such correlations represent only the average result obtained for a limited number of vehicles when operated under prescribed conditions.

Appendix: Volatility.—Change the second and third paragraphs to read as follows:

> Gasolines which vaporize too readily may boil in fuel lines or in carburetors and cause a decrease in the fuel flow to the engine, resulting in rough engine operation or stoppage (vapor lock). Conversely, gasolines which do not vaporize readily enough may cause hard starting and poor warm-up and acceleration, as well as unequal distribution of fuel to the individual cylinders. These conditions can be minimized by proper selection of volatility requirements, but cannot always be avoided. For example, during spring and fall a gasoline of volatility suitable for satisfactory starting at the frequent low temperatures encountered may be susceptible to vapor lock in some consuming units under some operating conditions during occasional hot periods.
>
> In the ASTM specifications volatility limits are established in terms of vapor pressure and distillation test results. Three types of automotive requirements are recognized: Type A, for the general run of automotive service; Type B, for service where cold starts and short trips are the chief consideration; and Type C, where hard, steady driving is the chief consideration. Provisions are made for three types of gasoline to conform to these three types of automotive requirements. In addition, provisions are made for three seasonal grades of each gasoline type. These grades differ with respect to the vaporization tendencies of the lighter fractions.

[2a] These methods will appear in the "ASTM Manual on Calibrating Liquid Containers," issued as a separate publication.

[3] 1955 Book of ASTM Standards, Part 5.

[3a] See Editorial Note on p. 280.

Appendix: Reid Vapor Pressure.—Replace this paragraph with the following:

Vapor pressure is the force exerted on the walls of a closed container by the vaporized portion of a liquid at a certain temperature; and conversely, it is the force which must be exerted on the liquid to prevent it from vaporizing further. Vapor pressure increases with temperature for any given gasoline. The ASTM Reid vapor pressure is measured at a temperature of 100 F in the presence of a volume of air which is four times the volume of liquid gasoline, and is expressed as pounds per square inch absolute.

The Reid vapor pressure of motor gasoline is the best single criterion of freedom from vapor lock in automotive equipment. When used in conjunction with the initial portion of the distillation curve the vapor lock relationships become more precise. In addition, these data are closely related to the composition of the gasoline in terms of the lightest hydrocarbons.

Appendix: Distillation.—Change to read as follows:

The ASTM distillation test provides a measure, in terms of volatility, of the relative proportions of all the hydrocarbon components of a gasoline. The ASTM specifications designate temperatures at which a minimum of 10 per cent, 50 per cent, and 90 per cent of the fuel shall be evaporated under the closely defined conditions of the prescribed test method. These distillation and vapor pressure characteristics define and control: starting, warm-up, carburetor icing, acceleration, vapor lock, crankcase dilution, and in part, fuel economy.

The 10 per cent evaporated temperature should be low enough to insure ready starting under normal temperature conditions, but not so low that vapor lock may be experienced. Generally it should be lower in the winter than in the summer, but it is obvious that gasoline used in vehicles such as buses, which are kept in warm garages in the winter and are never allowed to cool while in service, do not require gasoline with so low a 10 per cent evaporated temperature as do the majority of privately owned passenger cars.

Gasolines having the same 10 and 90 per cent evaporated temperatures may vary considerably in warm-up and acceleration quality because of differences in the boiling temperatures of the intermediate components or fractions. Good warm-up and acceleration properties can be assured by specifying, in addition, a suitable maximum limit for the 50 per cent evaporated temperature. The 90 per cent evaporated temperature should be low enough to preclude excessive dilution of the crankcase lubricating oil.

Appendix: Gum.—Change the second paragraph to read as follows:

A distinction should be made between gum and residues of oil which may appear on the gum test. Small amounts of oil are sometimes added to gasoline by manufacturers. Where such oil is present the ASTM Gum test cannot, of course, be considered a significant indication of gum content. In such cases an estimate of the gum content may be made as prescribed in the method.

D 613 – 48 T,[3] Test for Ignition Quality of Diesel Fuels by the Cetane Method:

It has been proposed that the present coincident-flash method of instrumentation be replaced by the Ignition-Delay Meter, Model ECS-1. Directions for effecting this revision are appended hereto.[4] To permit users of the ASTM-CFR Cetane Unit time to acquire this new equipment, these revisions will become officially effective on June 1, 1957.

D 614 – 49 T,[3, 5] Test for Knock Characteristics of Aviation Fuels by the Aviation Method:

Section 9(b).—Add the following sentence:

If a tendency for the fuel to bubble or boil in the fuel level sight glass is observed when making these adjustments, it is recommended that the carburetor cooling apparatus be used as described in Section 112(*a*) of Supplement I on Apparatus, and Section 323 of Supplement III on Operation. For normal conditions, however, carburetor cooling should not be used.

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[4] These revisions were accepted and incorporated in the revised method which appears in the 1956 Supplement to Book of ASTM Standards, Part 5, p. 133.

[5] ASTM Manual of Engine Test Methods for Rating Fuels, issued as a separate publication, October, 1952.

Supplement I:

Section 112(a).—Add the following after the last sentence:

A carburetor cooling attachment is available for testing fuels which give evidence of premature vaporization in the fuel induction system. This apparatus, shown in Fig. 2 (*Waukesha Service Bulletin No. 71*), is designed in two pieces: (*1*) to cool the horizontal jet and selector valve, and (*2*) to cool the No. 1 float bowl and tank.

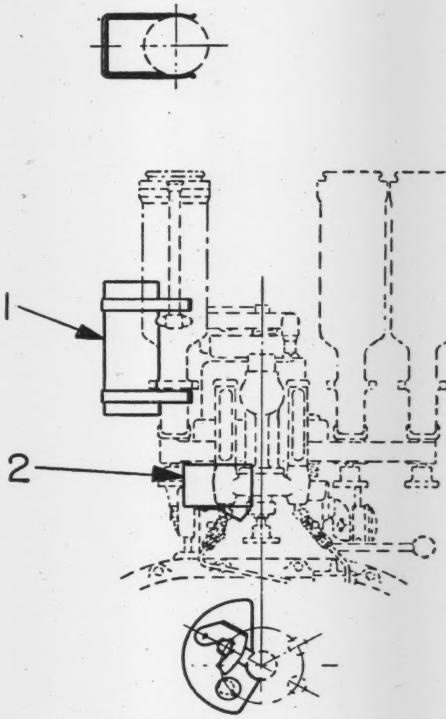
The jackets are made of stainless steel and are easily attached to or removed from the carburetor. When filled with dry ice, this equipment will permit ratings to be made in fuels of relatively high vapor pressure. The equipment will fit either the three or four bowl carburetor of the Motor, Research, and Aviation units.

Supplement III:

Section 323.—Add the following:

For testing fuels which give evidence of premature vaporization in the fuel induction system, the carburetor cooling attachment, described in Section 112(*a*) and shown in Fig. 2 (See accompanying Fig. 1), should be used. Premature vaporization may result in uneven engine operation and is often indicated by bubble formations, boiling, or abnormal level fluctuations of the fuel level in the sight glass. These conditions are not always apparent in the sight glass and often can be recognized only by erratic engine and knockmeter operation. Variables, such as ambient room temperature, engine location in the laboratory, and front end volatility of the fuel, are some of the reasons for variations in the ability of units to rate certain fuels. A positive statement regarding the use of cooling equipment cannot be made, other than cautioning operators to be observant and to use judgment in determining the necessity for carburetor cooling. Care should be exercised when rating fuels with high freezing point components, for temperatures reached in the carburetor may result in the separation of certain compounds. For normal conditions carburetor cooling is not necessary and is not recommended.

The tank cooling jacket is installed and held in place by spring arms which snap around the No. 1 tank and float bowl. The valve cooling jacket is installed and held in place by a cam latch which hooks around the No. 1 bowl drain valve. When used on the four bowl carburetor, the drain valve from No. 2 bowl will have to be removed to install the selector valve jacket. The valve can be replaced through the hole provided in the jacket. Nothing need be disturbed when applying either jacket to the three bowl carburetor on any of the methods.



1—Jacket for Cooling Horizontal Jet and Selector Value.

2—Jacket for Cooling the No. 1 Float Valve and Tank.

FIG. 1.—Carburetor Cooling Jackets.

D 721 - 55 T,[3] Test for Oil Content of Petroleum Waxes:

Section 3 (a).—Change to read as follows:

(*a*) *Filter Stick and Assembly,* consisting of a 10-mm diameter sintered glass filter stick of 10 to 15 μ maximum pore diameter as determined by the method in the Appendix, provided with an air pressure inlet tube and delivery nozzle. It is provided with a ground-glass joint to fit a 25 by 170-mm test tube. The dimensions for a suitable filtration assembly are shown in Fig. 1.

Note 1.—Revise by replacing the words "medium porosity" with the phrase "10 to 15 μ maximum pore diameter as determined by the method in the Appendix.

New Appendix.—Add an Appendix describing the method for obtaining the pore diameter of the apparatus in Section 3(*a*) as appended hereto.[2]

D 910 – 53 T,[3,3a] Specifications for Aviation Gasolines:

Table I.—In Footnote *d*: change "shall consist essentially of an alkyl-substituted anthraquinone" to "shall consist essentially of 1,4-dialkylamino anthraquinone."

D 1019 - 55 T,[3] Test for Olefinic Plus Aromatic Hydrocarbons in Petroleum Distillates:

Section 3(*c*).—Add the following after the first sentence: "The distance between the bottoms of opposite flasks when in the centrifuge in operating position shall be between 19½ and 22½ in."

Note 3.—Insert the following after the first sentence: "Since the distance between the bottoms of the cups in the hand centrifuge is less than in the usual motor-driven centrifuge, more than 3 min time of centrifuging may be required (see Section 6(*d*)). Centrifuging should be continued until constant volume is obtained."

Section 6(*d*).—Substitute "at 950 to 1100 rpm" for "at approximately 1000 rpm."

D 1085 - 52 T,[3] Method of Gaging Petroleum and Petroleum Products:

Section 5.—Add a new Paragraph (*e*), relettering the present Paragraphs (*e*) and (*f*) accordingly, to read as follows:

(*e*) *Incrustation.*—A tank used continually may accumulate deposits such as rust, wax, paraffin, tar, water, and sulfur on the inside of the shell and roof supports. Such incrustation decreases the capacity of the container and may cause inaccuracy in the determination of quantity. A thorough cleaning of a tank in this condition is necessary before accuracy may be obtained.

Section 6.—Change the last sentence to read: "Use the same gaging procedure and the same or matched equipment for both opening and closing gages."

Section 10.—Replace with the following:

(*a*) *Design:*

(*1*) Tanks having a variable vapor space, Fig. 4, are used principally for long-time storage of finished volatile stocks. The upward and downward movement of the roof with the expansion and contraction of the confined vapors largely eliminates breathing losses. In the breather-roof tank, the roof is a flexible steel diaphragm. In the balloon-roof tank, the diaphragm extends beyond the tank shell to provide a greater vapor space. The lifter-roof (gasometer-roof) tank is similar to the common gas holder in construction and operation. The roof rises and falls in a liquid seal.

(*2*) In addition to the types mentioned above, several varieties of tanks utilize fabric membranes to withhold the volatile vapors within a limited area. One type utilizes a hemisphere or small vertical tank on the top of an otherwise conventional cone-roof tank. The membrane is designed to fit the inside of the hemisphere (or tank) and to move up or down as the contents and/or vapor pressure of the material requires.

(*b*) *Gaging Equipment:*

(*1*) Tanks of the variable vapor space type described in Paragraph (*a*) Item (*1*) have either a gaging pipe (well) and manometer, Fig. 13, or a pressure lock, Fig. 6. It is recommended that the bottom of the gaging pipe be at least 12 in. above the bottom of the tank. If there is water or sediment in the tank, keep the level of this material at least 2 in. below the end of the gaging pipe. The manometer in the roof of the tank is used to determine the tank pressure or vacuum.

(*2*) For the type of system having a fabric membrane in a hemispherical or vertical cylindrical section attached to the roof (or in a container apart from the tank proper) gaging equipment similar to that described in Item (*1*) is provided.

D 1086 – 54 T,[3] Method of Measuring the Temperature of Petroleum and Petroleum Products:

Table II.—Insert the following preceding the "Angle Stem" thermometer:

Name	ASTM Thermometer	Range	Length, in.	Graduation	Accuracy
ASTM Tank.............	97 F-56 T	0 to 120 F	12	1 F	±0.5 F
ASTM Tank.............	98 F-56 T	60 to 180 F	12	1 F	±0.5 F

Section 8(a).—Insert the following after the first sentence: "Take temperatures at the same time the liquid level is gaged."

D 1160 – 52 T,[3] Test for Reduced Pressure Distillation of Petroleum Products:

Section 3(a).—Add the following new sentence: "An alternate column assembly[5] is shown in Fig. 10, Appendix."

Section 3(b).—Add a new sentence to read: "This heater is not to be used with the vacuum-jacketed column assembly. An alternate heater,[6] for use with the alternate flask and column assembly, is shown in Fig. 10, Appendix."

New Footnotes 5 and 6.—Add new Footnotes 5 and 6, renumbering the present Footnote 5 and its reference as Footnote 7, to read as follows:

[5] This column assembly may be obtained from the Glass Engineering Laboratories, Inc., at either Belmont, Calif., or 6309 South Broadway, Los Angeles 3, Calif., by specifying "Vacuum-Jacketed Distillation Head for ASTM Method D 1160."

[6] This heating assembly may be obtained from the Glas-Col Apparatus Co., 711 Hulman St., Terre Haute, Ind., by specifying "Quartz Heating Mantle, ASTM Method D 1160-B, 500 ml," and "Insulating Top, ASTM Method D 1160-T, 500 ml."

Section 4.—Letter the present wording as (*a*) and add a new Paragraph (*b*) after Note 2, to read as follows:

(*b*) *Reference Oil Check.*—Check the assembled apparatus, including the previously calibrated pressure measuring and thermocouple-potentiometer system, to indicate proper assembly and operating control. Conduct the test procedure as described at the test pressure in connection with a specific sample or at two or more pressures in connection with general checks of the equipment, using ASTM Cetane reference fuel.

If,

(*1*) The thermocouple and pressure measuring device have been properly calibrated,

(*2*) The thermocouple has been installed as directed in Section A2, Appendix,

(*3*) The distillation pressure is regulated within the allowable limits defined in Section 3 (*g*), and

(*4*) The construction of the column conforms with the specifications of Section A1(*b*), Appendix,

Then, the average of distillation temperatures obtained in the 10 to 90 per cent range, inclusive, should conform with the data in the following table:

Pressure, mm	Range of Average Temperature, deg Fahr
5.0..............	271.5 to 277.5
10.0..............	297.5 to 303.5
20.0..............	326.0 to 332.0
40.0..............	358.0 to 364.0
50.0..............	369.0 to 375.0

New Footnote 8.—Add a new Footnote 8 to read as follows:

[8] ASTM Cetane reference fuel is available from Humphrey & Wilkinson, Inc., Divine St., New Haven, Conn.

Footnotes 6 to 11.—Renumber the present Footnote 6 and its reference as Footnote 9, and renumber the present Footnotes 8 to 11 and their references accordingly.

New Note 3.—Add a new Note 3 after Section 4(*b*), renumbering the present Note 3 as Note 4, and subsequent Notes and references accordingly, to read as follows:

NOTE 3.—For pressures, P, over 5.0 mm not given in the table, the range of average temperature should not deviate by more than 3 F from a temperature, t, given by:

$$t, \text{deg Fahr} = \frac{3296.37}{7.03044 - \log_{10} P} - 246.15$$

Appendix I.—Designate as Appendix.

Footnote 12.—Delete the present Footnote 12 and its reference, and renumber subsequent footnotes and references accordingly.

Appendix II: Figure 1.—Renumber as Fig. 10 and transfer to the present Appendix I.

Appendix II.—Delete; and delete references to Appendix II in the text.

D 1220 – 55 T,[6] Methods for Calibrating Upright Tanks:

New Note 7.—Add a new Note 7 to the end of Section 13(*b*), renumbering subsequent notes and references accordingly, to read as follows:

NOTE 7.—Experience in checking the circumference measurements of bolted steel tanks indicates that such tanks, in sizes up to a nominal capacity of 1000 bbl, show no significant volumetric change over a period of 10 yr. Tanks having a nominal capacity in excess of 1000 bbl showed volumetric changes of a small order of magnitude over a 10-yr period which may be sufficient to warrant recalibration of the tank when considering the type of service and the value of the product involved.

Delete the procedures for calibrating spheres, spheroids, and pressure and non-pressure tank car tanks which were proposed and approved in 1955.

D 1269 – 53 T,[3] Method for Polarographic Determination of Tetraethyllead in Gasoline:

Section 5(a).—Replace with the following:

5. (*a*) Add the following to a 250-ml volumetric flask containing approximately 100 ml of distilled water: 25 ml of the standard lead solution, 2 ml of gelatin, 25 ml of cadmium solution, and 25 ml of concentrated hydrochloric acid. Dilute the mixture with distilled water to 250 ml and mix well.

D 1319 – 55 T,[3] Test for Hydrocarbon Types in Liquid Petroleum Products (Fluorescent Indicator Adsorption (FIA) Method):

Section 4(a).—Revise the first sentence to read as follows:

4. (*a*) *Silica Gel*, 100 to 200 mesh,[5] manufactured to insure minimum olefin polymerization and to conform to the sieve test requirements shown in Table I.

TABLE I.—SIEVE ACCEPTANCE TESTS OF SILICA GEL.

(Table I for D 1319)

Sieve Size[a]		Particles Passing Through Sieve, per cent by weight
Designation No.[a]	Microns	
80	177.........	100
100	149.........	95 min
200	74.........	15 max

[a] Detailed requirements for these sieves are given in ASTM Specifications E 11, 1955 Book of ASTM Standards, Part 5.

Table I.—Insert the accompanying Table I after Section 4(*a*).

New Note 5.—After Section 5 (*a*) add a new Note 5, renumbering the present Notes 5 and 6 and their references as Notes 6 and 7, to read as follows:

NOTE 5.—When the simultaneous preparation of a number of columns is desirable, it is convenient to hang a reasonable number from a frame or rack to which a vibrator is attached. Filling and packing may then proceed concurrently, provided that the equipment will give uniform packing of all tubes.

New Note 8.—Insert a new Note 8 after Section 5 (*d*), renumbering the present Note 7 as Note 9, to read as follows:

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[6] ASTM Manual on Calibrating Liquid Containers (Upright Tanks), issued as a separate publication, January, 1954.

NOTE 8.—An acceptable alternative method of introducing the sample is as follows: Chill the sample and a 1-ml hypodermic syringe to 35 to 40 F. Draw 0.75 cu cm of sample into the syringe and discharge with the tip inserted 20 to 30 mm below the surface of the gel in the charger section. No additional gel plug is needed, since the charger section is filled initially with a 40-mm section of gel.

Section 5(f).—Delete "always" from the last sentence and add the following phrase: "except that when no blue coloration is present, the brown or reddish ring shall be considered as part of the next distinguishable zone below it in the column."

WITHDRAWAL OF TENTATIVE

The committee recommends the withdrawal of the Tentative Method of Test for Steam Emulsion of Lubricating Oils (**D 157 - 51 T**),[3] subject to the adoption of the proposed new Tentative Method of Test for Emulsion Characteristics of Steam-Turbine Oils, which is appended hereto.[2]

REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommends that the Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (**D 95 - 46**)[3] be revised and reverted to tentative status. The revision consists of replacing the present text, with minor editorial changes, by the proposed method appended to Standard Method D 95 in 1955.[7]

ADOPTION OF TENTATIVES AS STANDARDS WITHOUT REVISIONS

The committee has reviewed the following four tentatives[3] which have stood two or more years without revision and which represent best present-day practices, and recommends that they be approved for reference to letter ballot of the Society for adoption as standards:

D 526 - 53 T, Test for Tetraethyllead in Gasoline.
D 972 - 51 T, Test for Evaporation Loss of Lubricating Greases and Oils,
D 1219 - 52 T, Test for Mercaptan Sulfur in Jet Fuels (Color-Indicator Method), and
D 1323 - 54 T, Test for Mercaptan Sulfur in Jet Fuels (Amperometric Method).

ADOPTION OF TENTATIVES AS STANDARDS WITH REVISIONS

The committee has reviewed the following three tentatives which represent best present-day practices, and recommends that they be approved for reference to letter ballot of the Society for adoption as standard with the revisions given below:

D 130 - 55 T,[3] Test for Copper Corrosion by Petroleum Products:

Section 4(a).—Change to read: "(*a*) *Wash Solvent*, use knock test grade *iso*octane."

Section 4(d).—Change "(Note 2)" to "(see Note 2)."

Section 5(a).—In the last sentence change the words "sulfur-free acetone" to "knock test *iso*octane."

Section 5(b).—In sentences one and two replace the word "acetone" with "*iso*octane." At the end of sentence five, add "(see Note 4)."

New Note 4.—Add a new Note 4 to follow Section 5(*b*), to read as follows:

NOTE 4.—It is important to polish the whole surface of the strip uniformly to obtain a uniformly stained strip. If the edges show wear (surface elliptical) they will likely show more corrosion than the center. The use of a vise will facilitate uniform polishing.

New Section 6.—Add a new Section 6 as follows:

6. (*a*) *Sample.*—It is particularly important that gasoline and fuel samples for this test be

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[7] **ASTM Standards on Petroleum Products and Lubricants, November, 1955.**

collected in clean dark bottles or cleaned tinned cans; to clean new tinned cans, rinse free of rosin or soldering flux with the sample to be tested. Care should be taken during sampling to protect the samples from exposure to direct or even diffused daylight (see Note 5). Make the test as soon as possible after sampling.

NOTE 5.—Short wavelengths of light (violet and ultraviolet) have a marked effect on reducing, or removing entirely, the coloration tendency of the corrosive sulfur present in the sample on copper.

(*b*) If suspended water (haze) is observed in the sample (see Note 6), dry by filtering a sufficient volume of sample through a medium rapid qualitative filter (for example, Whatman No. 4), into the prescribed clean, dry test tube. Carry out this operation in a darkened room or under a light-protected shield.

New Note 6.—Renumber, as Note 6, the present Note 4 and its reference, and have it follow the new Section 6(*b*).

Section 6.—Renumber as Section 7; and revise sentence two of Paragraph (*c*), changing "in sulfur-free acetone" to "in knock test *iso*octane." In sentence three, replace "dry with quantitative filter paper, and inspect" with "dry with quantitative filter paper (by blotting and not by wiping), and inspect." In the last sentence, change "staining it can be avoided if it is inserted in a flat test tube (Fig. 4)" to "staining can be avoided if it is inserted in a flat tube (see Fig. 4)."

Section 7.—Renumber as Section 8, and delete "excessively" from the last sentence of Paragraph (*e*).

Section 8.—Renumber as Section 9.

D 855 - 52 T,[3] Method of Analysis of Oil-Soluble Sodium Petroleum Sulfonates:

Note 3.—Change to read as follows:

NOTE 3.—It is important to adhere as closely as possible, to the quantities and concentrations of isopropyl alcohol prescribed in Sections 5 and 6(*a*). If, for example, the volume of titrant B exceeds 3 ml, a volume of 99 per cent isopropyl alcohol, equivalent to the excess, is added to the combined alcoholic solution. Final concentration in a separatory funnel should not be less than 48 per cent nor more than 52 per cent by volume of isopropyl alcohol.

Section 14.—In the tabulations in Paragraphs (*a*) and (*b*) omit the three lines: "Inorganic salts, weight per cent," "Below 1.00," and "Above 1.00."

D 1216 - 52 T,[3] Method of Analysis of Calcium and Barium Petroleum Sulfonates:

Section 16(a).—Delete "Inorganic salts, weight per cent . . . 0.09" from the table of constituents.

Section 16(b).—Delete "Inorganic salts, weight per cent . . . 0.362" from the table of constituents.

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends immediate adoption of revisions to the eight standards listed below and accordingly asks for a favorable nine-tenths vote at the Annual Meeting in order that these revisions may be referred to letter ballot of the Society:

D 56 - 52,[3] Test for Flash Point by Tag Closed Tester:

The method has been extensively revised editorially and includes a new Section 8 on precision. The revised method is appended hereto.[2]

D 86 - 54,[3] Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products:

Section 3(d).—In the first sentence change "shall be fitted tightly" to read "shall be cooled to 55 to 65 F, and fitted tightly." Delete the last sentence.

Section 5.—Delete this section, renumbering Section 6 as Section 5.

New Section 6.—Add a new section, to read as follows:

6. *Precision.*—The following criteria, based on 95 per cent probability, should be used for

judging the acceptability of results:

(*a*) Duplicate results obtained by the same operator and apparatus should be considered suspect if they differ by more than the repeatability which, according to Fig. 3, is appropriate to the test result obtained and to the rate of change in thermometer reading which prevailed at the stage at which the result was obtained.

(*b*) The results obtained by each of two laboratories should be considered suspect if they differ by more than the reproducibility which, according to Fig. 3, is appropriate to the test result obtained and to the rate of change in thermometer reading which prevailed at the stage at which the result was obtained.

(*c*) To facilitate the use of Fig. 3, the rate of change in thermometer reading in degrees Fahrenheit per the percentage evaporated or recovered, at any point between the initial boiling point and the end point or dry point, should be assumed to be the same as the average rate between two data points which are equidistant above and below the point in question, provided that the span from the point in question to either of the other data points does not represent more than 10 per cent evaporated or recovered in any case, nor more than 5 per cent if the point in question is not included in the 10 to 90 per cent range. For the initial boiling point, end point, or dry point, the rate of change should be assumed to be the same as the average rate over an interval, not to exceed 5 per cent evaporated or recovered, between the extreme point and the next data point above or below it.

New Fig. 3.—Add the accompanying Fig. 3.

D 88 – 53,[3] Test for Viscosity by Means of the Saybolt Viscometer:

This method has been extensively revised editorially. In addition, a special procedure for waxy or resinous materials has been included. A copy of the revised and enlarged method is appended hereto.[2]

D 92 – 52,[3] Test for Flash and Fire Points by Means of Cleveland Open Cup:

The method has been extensively revised editorially and includes a new Section 7 on precision. The revised method is appended hereto.[2]

D 323 – 55,[3] Test for Vapor Pressure of Petroleum Products (Reid Method):

Section 1.—Change to read as follows:

1. This method of test is for the determination of the absolute vapor pressure of volatile crude oil and volatile non-viscous petroleum products, except liquefied petroleum gases.

NOTE 1.—Because the external atmospheric pressure is counteracted by the atmospheric pressure initially present in the air chamber, the "Reid vapor pressure" is approximately the vapor pressure of the material at 100 F in pounds per square inch absolute.

Section 2.—Change the first sentence to read: "The gasoline chamber of the vapor pressure apparatus is filled with the chilled sample and connected to the air chamber at 100 F or other temperature."

Change the third sentence to read: "The 'manometer reading' corresponding to the pressure, read on the gage attached to the apparatus is the Reid vapor pressure suitably corrected (Table I) if the air chamber was initially at a temperature other than 100 F."

In the second paragraph of Section 2, change "for a partial air-saturation for products with Reid vapor pressures below" to "for partial air saturation of products with Reid vapor pressure below."

Notes 1 and 2.—Renumber as Notes 2 and 3, respectively.

Section 3(e).—Change the paragraph heading to "(*e*) *Thermometer for Water Bath and 100 F Air Chamber Procedure.*"

Section 3(f).—Change to read as follows:

(*f*) *Air Chamber Thermometer.*—When the ambient temperature procedure is employed, a thermometer conforming to the following requirements shall be used: Length, approximately 12 in.; range, −40 or −30 F to +120 or +130 F; graduated in 1 F divisions; total immersion; scale error not greater than 1 F.

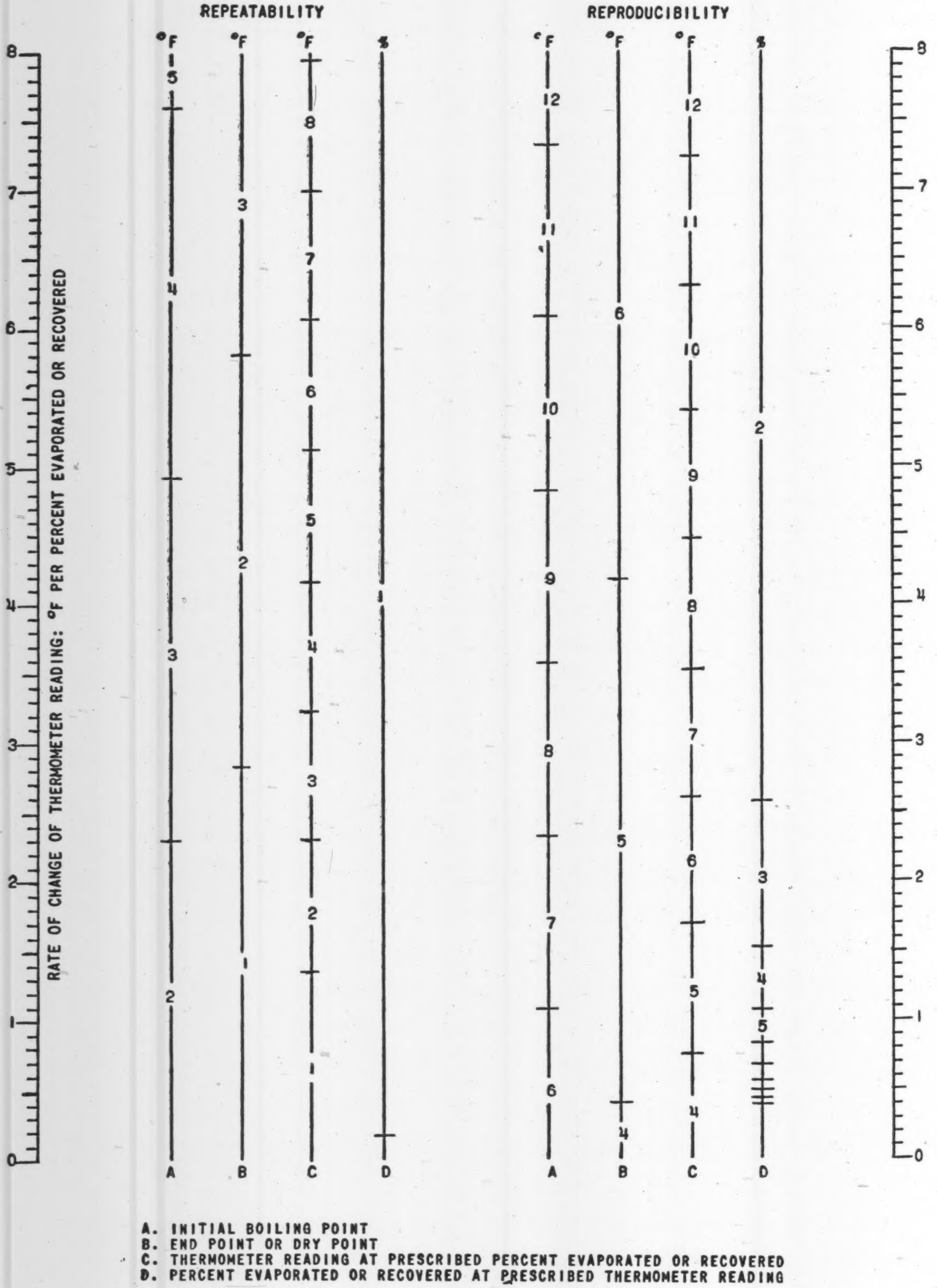

FIG. 3.—Precision of Distillation Method D 86.

Section 5.—Change to read as follows:

(*c*) *Preparation of Air Chamber* (*100 F Procedure*).—After purging and rinsing the air chamber and pressure gage in accordance with Section 6(*e*), connect the gage to the air chamber. Immerse the air chamber to at least 1 in. above its top in the water bath (see Note 5), for not less than 10 min just before coupling it to the gasoline chamber. Do not remove the air chamber from the bath until the gasoline chamber has been filled with sample as described in Section 6(*a*).

(*d*) *Preparation of Air Chamber* (*Ambient Temperature Procedure*).—As an alternate to Paragraph (*c*), the air chamber may be adjusted to ambient or other temperature which may be determined with an accuracy of at least 1 F in the following manner. After purging and rinsing the air chamber and pressure gage in accordance with Section 6(*e*), connect the gage to the air chamber. Insert the thermometer into the air chamber, supporting it by means of a loosely-fitting (not air-tight) stopper in the opening of the air chamber. Adjust the position of the thermometer so that it is aligned as closely as possible with the axis of the air chamber, and with the thermometer bulb located in the air chamber, about 9 in. from the opening. Leave the thermometer in position until the temperature reading has remained constant within 1 F for a period of 5 min or more just before coupling the air chamber to the gasoline chamber. At this time, record the thermometer reading as the "initial air temperature."

Note 3.—Delete this note.

Section 6(*b*).—Change to read as follows:

(*b*) *Assembly of Apparatus.*—Without delay, and as quickly as possible, attach the air chamber to the gasoline chamber. Not more than 20 sec shall be consumed in completing the assembly of the apparatus after filling the gasoline chamber, using the following sequence of operations:

(*1*) Refill the gasoline chamber to overflowing,

(*2*) Read the "initial air temperature" or remove the air chamber from the water bath, and

(*3*) Connect the air chamber to the gasoline chamber.

Section 6(*c*).—Add the following after the last sentence: "When at any time a leak is detected, discard the test."

Note 4.—Delete the last sentence.

Section 6(*d*).—Revise the last sentence to read: "Immediately remove the pressure gage and check its reading against that of the manometer, recording the value found as the Reid vapor pressure (under the procedure of Section 5(*c*)), or as the "manometer reading" to be used in the calculations of Section 8 (under the procedure of Section 5(*d*))."

Note 5.—Change "if this purging is done" to "if the purging of the air chamber is done."

Section 7(*f*).—Revise to read: "(*f*) *Coupling the Apparatus.*—Carefully observe the requirements of Section 6(*b*)."

Section 7(*h*).—Revise the second sentence to read: "Be certain that the temperature of the air in the air chamber at the time of coupling with the gasoline chamber (Section 6(*b*)) has remained constant within 1 F for a period of 5 min or more."

Section 8.—Change to read as follows:

8. (*a*) *Change in Pressure of Water Vapor and Air.*—For the ambient temperature procedure set forth in Section 5(*d*), calculate the "Reid vapor pressure" of the sample under test by applying to the "manometer reading" the correction given in Table I for the change in pressure of the water vapor and air in the chamber on heating from the "initial air temperature" to 100 F.

(*b*) *Recording Results.*—If the 100 F Procedure was employed (Section 5(*c*)), record the result observed in Section 6(*d*) as the "Reid vapor pressure" in pounds without reference to temperature or to unit of surface. If the ambient temperature procedure (Sections 5(*c*) and 8(*a*)) was used, record the value resulting from the application of the correction from Section 8(*a*) as the "Reid vapor pressure" without reference to temperature or unit of surface.

Note 6.—Delete, renumbering the subsequent notes and their references accordingly.

Table I.—Replace with the following Table I:

TABLE I.—CORRECTIONS TO BE SUBTRACTED FROM "MANOMETER READINGS" FOR CALCULATING REID VAPOR PRESSURE.

(Table I, Method D 323)

Initial Air Temperature,[a] deg Fahr	Barometric Pressure,[a] mm		
	760	700	600
32	2.90	2.70	2.45
40	2.60	2.45	2.20
50	2.20	2.10	1.90
60	1.80	1.70	1.55
70	1.40	1.30	1.20
80	0.95	0.90	0.85
90	0.50	0.50	0.45
100	0.00	0.00	0.00
110	−0.55	−0.55	−0.50

[a] For other temperatures and pressures, the corrections may be calculated by means of the following equation:

$$\text{Correction} = \frac{(P - P_t)\,(t - 100)}{460 + t} - (P_{100} - P_t)$$

where:

t = air chamber temperature at beginning of test, in degrees Fahrenheit,

P = barometric pressure, in pounds per square inch, at time of test (if a barometer is not available, the normal barometric pressure may be used),

P_t = vapor pressure of water, in pounds per square inch, at t deg Fahr, and

P_{100} = vapor pressure of water in pounds per square inch, at 100 F = 0.95.

Calculated corrections are to be rounded off to the nearest 0.05 lb.

Example: In line 6 change 745 mm to 700 mm.
In lines 7 and 9 change 0.95 lb to 0.90 lb.
In line 11 change 10.55 lb to 10.60 lb.

Section 9(a).—In the sixth line change "8" to "6(*d*) and 8(*a*)."

Section 13(c).—Replace the second sentence with the following:

Not more than 25 sec shall be consumed in completing the assembly of the apparatus after filling the gasoline chamber, using the following sequence of operations: (*1*) read the initial air temperature or remove the air chamber from the water bath, (*2*) connect the air chamber to the gasoline chamber, and (*3*) open the gasoline chamber ½-in. valve.

Section 15(d).—Change to read as follows:

(*d*) *Air Chamber Temperature.*—The provisions of Section 5(*c*) shall be followed; the provisions of Section 5(*d*) shall not apply.

D 357 – 53,[3, 8] Test for Knock Characteristics of Motor Fuels by the Motor Method:

Section 11(b).—Add the following after the last sentence:

If a tendency for the fuel to bubble or boil in the fuel level sight glass is observed when making these adjustments, it is recommended that the carburetor cooling apparatus be used as described in Section 112, Appendix I on Apparatus, and Section 317, Appendix III on Operation. For normal conditions, however, carburetor cooling should not be used.

Appendix I: Section 112.—Revise as shown in this report under proposed revisions to ASTM Method D 614.[9]

Appendix III: Section 312.—Revise as shown in this report under proposed revisions to ASTM Method D 614.[9]

D 908 – 55,[3, 9] Test for Knock Characteristics of Motor Fuels by the Research Method:

Section 11(b).—Add the following after the last sentence:

If a tendency for the fuel to bubble or boil in the fuel level sight glass is observed when making these adjustments, it is recommended that the carburetor cooling apparatus be used as described in Section 112, Appendix I on Apparatus, and Section 323, Appendix III on Operation. For normal conditions, however carburetor cooling should not be used.

Appendix I: Section 112.—Revise as shown in this report under proposed revisions to ASTM Method D 614.[9]

Appendix III: Section 312.—Revise as shown in this report under proposed revisions to ASTM Method D 614.[9]

D 1250 – 55, ASTM-IP Petroleum Measurement Tables:

Withdraw the following extensions to Tables 6 and 7 which were proposed in 1955:

(*1*) Low-Temperature Table (0 to −50 F)

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[8] ASTM Manual for Rating Motor Fuels by Motor and Research Methods, issued as a separate publication, 1956.

[9] See p. 268.

for Volume Reduction to 60 F (extension of Table 6).

(*2*) Low-Temperature Table (0 to −50 F) for Volume Reduction to 60 F, abridged (extension of Table 7).

TENTATIVES CONTINUED WITHOUT REVISION

The committee has reviewed the following 13 tentatives[3] which have stood two years or more without revision and, for good and sufficient reasons, discussed within the committee, recommends that they be continued as tentative without revision:

D 96 - 52 T, Test for Water and Sediment by Means of Centrifuge,

D 155 - 45 T, Test for Color of Lubricating Oil and Petrolatum by Means of ASTM Union Colorimeter,

D 285 - 54 T, Test for Distillation of Crude Petroleum,

D 396 - 48 T, Specifications for Fuel Oils,

D 524 - 52 T, Test for Carbon Residue of Petroleum Products (Ramsbottom Coking Method),

D 808 - 52 T, Test for Chlorine in Lubricating Oils and Greases by the Bomb Method,

D 909 - 49 T, Test for Knock Characteristics of Aviation Fuels by the Supercharge Method,

D 1087 - 52 T, Recommended Practice for Volume Calculations and Corrections in the Measurement of Petroleum and Petroleum Products,

D 1091 - 54 T, Test for Phosphorus in Lubricating Oils, Lubricating Oil Additives, and their Concentrates,

D 1218 - 52 T, Test for Refractive Index and Refractive Dispersion of Hydrocarbon Liquids,

D 1317 - 54 T, Test for Chlorine in Lubricating Oil (Sodium Alcoholate Volumetric Method),

D 1318 - 54 T, Test for Sodium in Residual Fuel Oil by Flame Photometer, and

D 1322 - 54 T, Test for Smoke Point of Jet Fuels.

AMERICAN STANDARDS

The committee recommends the following for action by ASA Sectional Committee Z11 on Petroleum Products and Lubricants:

Approval as American Standard:

Test for Copper Corrosion by Petroleum Products (D 130 - 55 T), and

Test for Evaporation Loss of Lubricating Greases and Oils (D 972 - 51 T).

Reapproval as American Standard as Revised in This Report:

Test for Flash Point by Tag Closed Tester (D 56 - 52; Z11.24—1952),

Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (D 86 - 54; Z11.10—1955);

Test for Viscosity by Means of the Saybolt Viscosimeter (D 88 - 53; Z11.2–1953),

Test for Flash and Fire Points by Means of Cleveland Open Cup (D 92 - 52; Z11.6—1952),

Test for Water in Petroleum Products and Other Bituminous Materials (D 95 - 46; Z11.9–1947),

Test for Vapor Pressure of Petroleum Products (Reid Method) (D 323 - 55; Z11.44–1955),

Test for Knock Characteristics of Motor Fuels by the Motor Method (D 357 - 53; Z11.37—1953),

Test for Knock Characteristics of Motor Fuels by the Research Method (D 908 - 55; Z11.69—1953),

ASTM-IP Petroleum Measurement Tables (D 1250 - 55; Z11.83—1953; IP200/52), and

Test for Tetraethyllead in Gasoline (D 526 - 53 T; Z11.48—1953).

Method of Test for Saponification Number of Petroleum Products by Color-Indicator Titration (Tentative) (D 94 - 56 T; Z11.20)

Method of Test for Oil Content of Petroleum Waxes (Tentative) (D 721 - 56 T; Z11.52)

Method of Test for Olefinic Plus Aromatic Hydrocarbons in Petroleum Distillates (Tentative) (D 1019 - 56; Z11.71)

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[10]

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[10] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

This report has been submitted to letter ballot of the committee, which consists of 126 voting members; 71 members returned their ballots, of whom 65 voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

H. M. SMITH,
Acting Chairman.

W. T. GUNN,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-2 presented to the Society through the Administrative Committee on Standards the following recommendation:

Revision of Tentative Specifications for:

Gasoline (D 439 - 56 T), and
Aviation Gasolines (D 910 - 56 T).

These recommendations were accepted by the Standards Committee on September 5, 1956, and the revised specifications appear in the 1956 Supplement to Book of ASTM Standards, Part 5.

REPORT OF SECTION III ON OXIDATION OF TURBINE OILS, TECHNICAL COMMITTEE C ON TURBINE OILS

Turbine operators for some time have wanted a fast, reliable means of determining the condition of their turbine oils. In particular, they are interested in detecting early indications of oil oxidation. This need was brought to the attention of Technical Committee C on Turbine Oils by a representative of the industrial power companies. As expressed by that representative, the power companies believed in the reliability of ASTM Method D 943, Test for Oxidation Characteristics of Inhibited Steam-Turbine Oils,[1] but thought that it took too much time for a routine test. Also, the equipment required by Method D 943 was somewhat more complex and costly than was considered desirable by the smaller laboratories. Other nonstandard tests were available, but their dependability had not been established.

This problem was discussed at a special meeting of Technical Committee C in September, 1952. At that meeting it was decided to develop an oxidation test which would enable any small laboratory to determine quickly whether or not a used turbine oil was liable to "break" if continued in service for an additional year.

With that goal in mind, 13 laboratories participated in a preliminary study designed to include as many as practical of the various types of oxidation tests available. The following types of tests were included:

Test	Conditions
Dornte Oxygen	Several modifications conducted at 250 or 302 F for up to 310 hr
Modified Continental Oxidation	239 F, up to 1060 hr
Modified Staeger	230 F, up to 720 hr
Modified Brown Boveri	230 F, 96 hr
Sun Oxidation	280 F, 96 hr
IP Method 114/47	230 F, 90 hr
Rotary Bomb	302 F, up to 16 hr
Sligh Oxidation	392 F, 2½ hr
B-10 Oxidation	260 F, 40 hr

The results of this study narrowed the field to two or three types of tests. When the over-all simplicity and availability of equipment were considered, one method was selected for more intensive investigation. This was the B-10 Oxidation Test, hereinafter to be called the Short-Term Oxidation Test for Turbine Oils (STOT). A complete description of the method is given in Appendix I. Other methods of test are under continuing investigation by the committee.

Since the 40 hr required to complete this test seemed unduly long, the test was shortened arbitrarily to 6 hr, but no other significant changes were made in the procedure. With the modified method, new and used inhibited turbine oils and one uninhibited oil of turbine-oil quality were tested cooperatively. At the same time, one laboratory tested the same oils by ASTM Method D 943, the standard method. There is presented in Tables I and II the data obtained to date in the two cooperative testing programs of the short-time oxidation test (STOT) and ASTM Method D 943.

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[1] 1955 Book of ASTM Standards, Part 5.

TABLE I.—SUMMARY OF TEST RESULTS BY SHORT-TIME OXIDATION TEST (STOT) AND ASTM METHOD D 943 ON SERIES X OILS.

Sample No.	Short-Time Oxidation Test									
	Oil Company "A"		Oil Company "B"		Oil Company "C"		Oil Company "D"		Oil Company "E"	
	Neutralization Value (Increase)	Lead Loss, mg	Neutralization Value (Increase)	Lead Loss, mg	Neutralization Value (In crease)	Lead Loss, mg	Neutralization Value (Increase)	Lead Loss, mg	Neutralization Value (Increase)	Lead Loss mg
New oil 1	nil	nil	...	not reported	0.01	0.9	0.01	0.5	0.1	0.4
	nil	0.4	0.26				0.01	0.7		
Used oil 1 (20 000 hr)	0.02	1.0	0.03		0.03	1.7	0.01	0.7	0.03	1.3
	nil	1.1	0.33				0.00	0.6		
New oil 2	nil	1.6	0.01		0.01	2.3	nil	2.1	nil	2.0
	nil	2.4	0.32		0.00	2.8	nil	2.2		
Used oil 2 (26 000 hr)	nil	0.3	0.34		0.02	1.3	0.00	1.2	nil	1.3
	nil	0.8	0.24				0.01	1.1	...	
New oil 3	nil	nil	0.02[b]		0.02	0.9	0.01	0.7	0.01	0.7
	nil	0.2	0.14[b]				0.01	1.0		
			0.24							
Used oil 3 (45 000 hr)	nil	nil	0.10[b]		0.02	0.1	0.01	0.5	0.03	1.0
	nil	0.8	0.22[b]				0.01	0.9		
			0.26							
New oil 4	nil	0.1	...[b]		0.03	0.9	nil	2.1	0.01	0.6
	nil	0.8	0.14[b]		0.00	0.9	nil	2.5		
			0.26							
Used oil 4 (60 000 hr)	3.37	62.9	1.63		2.34	45.0	2.31	43.2	2.13[c]	47.3
	1.87	45.4	2.03		3.21	8.7	2.11	55.3		
New oil 5	0.01	0.2	...[b]		0.00	1.5	nil	3.2	nil	0.9
	nil	1.0	0.00[b]				nil	3.5		
			0.24							
Used oil 5 (50 000 hr)	nil	nil	...[b]		0.01	1.3	0.00	1.4	0.01	1.0
	nil	0.8	...[b]				nil	3.1		
New oil 6 (uninhibited)	0.72[a]	11.8	0.89[b]		1.32	26.2	0.68	9.4	1.17[c]	22.3
	0.29	5.3	2.98[b]				1.36	9.9		
			2.89							

[a] These tests were run several weeks apart.

[b] Wet air.

[c] Composite of quadruplicate runs.

TABLE I.—SUMMARY OF TEST RESULTS BY SHORT-TIME OXIDATION TEST (STOT) AND ASTM METHOD D 943 ON SERIES X OILS (*Concluded*).

Sample No.	Short-Time Oxidation Test						ASTM Method D 943			
	Oil Company "F"		Oil Company "G"		Oil Company "H"					
	Neutralization Value (Increase)	Lead Loss. mg	Neutralization Value (Increase)	Lead Loss, mg	Neutralization Value (Includes)	Lead Loss, mg	Hours in Test	Neutralization Value, mg of KOH per g		Status of Test
								Initial	Final	
New oil 1	0.0	0.4	nil nil	0 0.1	0.03[d]	5.8 0.9	1344	0.11	3.13	Terminated
Used oil 1 (20 000 hr)	0.0	0.4	nil nil	1.5 0.1	0.07	2.4 1.5	672	0.16	3.02	Terminated
New oil 2	0.0	2.7	nil nil	1.4 1.2	0.01	1.8 3.8	1344	0.11	5.94	Terminated
Used oil 2 (26 000 hr)	0.0	1.7	0.05 0.05	0.8 0.5	0.06	2.8 2.1	672	nil	4.90	Terminated
New oil 3	0.01	0.3	0.05 0.05	1.0 1.1	0.06	3.4 1.2	3024+	trace	0.11	Test discontinued
Used oil 3 (45 000 hr)	0.0	1.1	0.05 0.02	0.9 1.3	0.04	3.2 1.2	3024+	nil	0.27	Test discontinued
New oil 4	0.0	1.5	0.08 0.05	0.9 1.1	nil	1.6 1.2	2016	nil	2.86	Terminated
Used oil 4 (60 000 hr)	2.32	31.1	1.14 1.24	27.6 30.1	1.89	37.7 52.9	48	0.77	2.53	Terminated
New oil 5	0.0	1.4	0.03 0.03	1.3 1.5	0.03	1.9 1.6	3024+	nil	0.11	Test discontinued
Used oil 5 (50 000 hr)	0.0	1.2	0.02 nil	1.5 0.9	0.06	1.2 1.3	3024+	nil	0.11	Test discontinued
New oil 6 (uninhibited)	0.55	7.3	1.11 1.06	16.5 5.1	0.85	19.4 13.6	48	nil	3.35	Terminated

[d] Average of duplicate tests.

TABLE II.—SUMMARY OF RESULTS BY SHORT-TIME OXIDATION METHOD (STOT) AND ASTM METHOD D 943 ON SERIES XI OILS.

Sample No.	Short-Time Oxidation Test															
	Oil Company "A"				Oil Company "B"				Oil Company "D"				Oil Company "E"			
	Neutralization Value			Lead Loss, mg	Neutralization Value			Lead Loss, mg	Neutralization Value			Lead Loss, mg	Neutralization Value			Lead Loss, mg
	Initial	Final	Increase		Initial	Final	Increase		Initial	Final	Increase		Initial	Final	Increase	
Used oil 2A (2800 hr)	0.05	0.05	0.00	0.6	0.05	0.06	0.01	2.0	0.07	0.06	nil	1.7	0.04	0.06	0.02	1.4
	0.05	0.10	0.05	0.8					0.07	0.06	nil	1.5	0.04	0.06	0.02	1.0
Used oil 4A (3400 hr)	0.05	0.05	0.00	1.9	0.05	0.05	0.00	1.4	0.06	0.05	nil	1.9	0.04	0.04	0.00	1.3
	0.05	0.05	0.00	2.2					0.06	0.06	0.00	1.5	0.04	0.04	0.00	1.3
Used oil 6A (53 000 hr)	0.05	0.05	0.00	1.3	0.03	0.05	0.02	1.1	0.05	0.04	nil	1.1	0.04	0.04	0.00	0.7
	0.05	0.05	0.00	1.7					0.05	0.05	0.00	1.2	0.04	0.04	0.00	0.3
New oil 101	0.05	0.05	0.00	2.6	0.05	0.05	0.00	2.0	0.06	0.03	nil	1.6	0.04	0.04	0.00	1.6
	0.05	0.05	0.00	2.1					0.06	0.01	nil	1.6	0.04	0.04	0.00	0.9
Used oil 102 (54 000 hr)	0.10	0.10	0.00	1.8	0.05	0.11	0.06	2.8	0.10	0.08	nil	3.0	0.14	0.10	nil	2.3
	0.10	0.10	0.00	2.0					0.10	0.08	nil	3.2	0.14	0.12	nil	1.6
New oil 103	0.05	0.05	0.00	1.6	0.03	0.05	0.02	2.2	0.03	0.01	nil	1.7	0.02	0.04	0.02	1.5
	0.05	0.05	0.00	1.3					0.03	0.01	nil	1.6	0.02	0.02	0.00	0.2
Used oil 104 (111 309 hr)	0.25	0.25	0.00	1.3	0.17	0.17	0.00	3.5	0.22	0.21	nil	3.8	0.22	0.22	0.00	1.1
	0.25	0.25	0.00	2.4					0.22	0.21	nil	4.1	0.22	0.22	0.00	1.2
New oil 105	0.05	0.05	0.00	1.9	0.05	0.05	0.00	1.7	0.05	0.03	nil	1.8	0.04	0.02	nil	1.0
	0.05	0.05	0.00	2.5					0.05	0.02	nil	2.1	0.04	0.04	0.00	1.1
Used oil 106 (8000 hr)	0.10	0.10	0.00	1.2	0.05	0.05	0.00	0.9	0.05	0.02	nil	0.9	0.06	0.06	0.00	0.9
	0.10	0.10	0.00	1.4					0.05	0.02	nil	1.3	0.06	0.06	0.00	0.0

Note.—Oil companies "C" and "G," which submitted data on Series X oils, did not participate in tests on Series XI oils.

TABLE II.—SUMMARY OF RESULTS BY SHORT-TIME OXIDATION METHOD (STOT) AND ASTM METHOD D 943 ON SERIES XI OILS (*Concluded*).

Sample No.	Short-Time Oxidation Test								ASTM Method D 943			
	Oil Company "F"				Oil Company "H"				Hours in Test	Neutralization Value, mg of KOH per g		Status of Test
	Neutralization Value			Lead Loss, mg	Neutralization Value			Lead Loss, mg				
	Initial	Final	Increase		Initial	Final	Increase			Initial	Final	
Used oil 2A (2800 hr)	0.05	0.07	0.02	0.6	0.06	0.08	0.02	0.9	504	0.05	3.30+	Terminated
	0.05	0.05	0.00	1.3	0.06	0.08	0.02	1.5				
Used oil 4A (3400 hr)	0.05	0.05	0.00	1.7	0.04	0.07	0.03	1.8	672	0.05	4.40+	Terminated
	0.05	0.05	0.00	1.3	0.04	0.07	0.03	2.1				
Used oil 6A (53 000 hr)	0.05	0.05	0.00	0.7	0.03	0.05	0.02	1.0	3192	0.08	0.11	Discontinued—lack of sample
	0.05	0.05	0.00	0.5	0.03	0.05	0.02	1.2				
New oil 101	0.05	0.05	0.00	1.3	0.04	0.04	0.00	1.1	3192	0.05	0.44	Discontinued—lack of sample
	0.05	0.05	0.00	1.2	0.04	0.04	0.00	1.1				
Used oil 102 (54 000 hr)	0.07	0.07	0.00	1.6	0.09	0.10	0.01	2.3	1680	0.05	4.95	Terminated
	0.07	0.07	0.00	1.9	0.09	0.13	0.04	2.4				
New oil 103	0.02	0.02	0.00	0.1	0.02	0.04	0.02	1.3	1680	0.05	6.43	Terminated
	0.02	0.02	0.00	0.1	0.02	0.04	0.02	1.3				
Used oil 104 (111 309 hr)	0.16	0.18	0.02	0.5	0.14	0.15	0.01	2.5	672	0.22	6.60+	Terminated
	0.16	0.16	0.00	1.0	0.14	0.15	0.01	2.0				
New oil 105	0.09	0.10	0.01	1.2	0.04	0.04	0.00	1.8	1848	0.05	5.06	Terminated
	0.09	0.10	0.01	1.1	0.04	0.04	0.00	1.0				
Used oil 106 (8000 hr)	0.05	0.05	0.00	0.2	0.08	0.09	0.01	0.8	1848	0.05	5.94	Terminated
	0.05	0.05	0.00	0.1	0.08	0.10	0.02	0.7				

The committee now is about to start a third series of cooperative tests. Throughout the last two series, all the samples represent oils actually in service. Some of them are the same oils tested in the first series, modified only by the additional months and years of use the products have undergone. Thus, Technical Committee C is attempting to develop not just another oxidation test, but one which will reflect changes due to daily service.

The committee is aware that the usefulness of the short method can be established thoroughly only if it is widely known and used. Therefore, it is offering the procedure at this time to the public in the hope that its utility can be more quickly explored. The comments of users of the method are invited.

Respectfully submitted on behalf of the section,

E. A. REEHL,
Chairman.

REPORT OF COMMITTEE D-3

ON

GASEOUS FUELS*

Committee D-3 on Gaseous Fuels and its seven subcommittees held a joint meeting in New York, N. Y., on May 26, 1955.

R. H. Murray was elected secretary of the committee to replace K. R. Knapp upon his retirement shortly thereafter. The committee accepted the resignation of S. W. Burdick and E. F. Schmidt. J. F. Anthes was elected to the Advisory Committee. E. F. Hammerschmidt succeeded Mr. Burdick as chairman of Subcommittee VI.

The officers elected for the ensuing term of two years are as follows:

Chairman, L. T. Bissey.
Vice-Chairman, F. E. Vandaveer.
Secretary, R. H. Murray.

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the ollowing one tentative method be approved for reference to letter ballot of the Society for adoption as standard without change:

Tentative Method of:

Test for Sulfur in Fuel Gases (**D 1072 - 54 T**).[1]

This recommendation has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

AMERICAN STANDARDS

Approval as American Standard was accorded by the American Standards Association to the following four ASTM standards prepared by Committee D-3. The date of approval was August 12, 1955, and the ASA numbers assigned to the standards are indicated below:

Standard Methods of:

Analysis of Natural Gases by the Volumetric Chemical Method (**ASTM D 1136 - 53; ASA Z77.1—1955**),

Analysis of Natural Gases and Related Types of Gaseous Mixtures by the Mass Spectrometer (**ASTM D 1137 - 53; ASA Z77.2—1955**),

Test for Water Vapor Content of Gaseous Fuels by Measurement of Dew Point Temperature (**ASTM D 1142 - 53; ASA Z77.3—1955**), and

Sampling Natural Gas (**ASTM D 1145 - 53; ASA Z77.4—1955**).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee V on Determination of Special Constituents of Gaseous Fuels (L. T. Bissey, chairman) recommended the adoption as standard of Method D 1072 - 54 T referred to earlier in the report. Preliminary studies are in progress on the determination of particulate substances in fuel gases.

Subcommittee VI on Determination of Water Vapor Content of Gaseous Fuels (E. G. Hammerschmidt, chairman) plans to continue experimental work on the Brickell method for determination of water vapor in fuel gases.

Subcommittee VII on Complete Analysis of Chemical Composition of Gaseous Fuels (D. V. Kniebes, chairman) recommended the continuation as tentative of Method D 1302 - 53 T because of the objections of Committee E-14 on Mass Spectrometry to acceptance in its present form. This method is being rewritten in accordance with the recommendations of Committee E-14.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] 1955 Book of ASTM Standards, Part 5.

[2] The letter ballot vote on this recommendation was favorable; the results of the vote are on record at ASTM Headquarters.

This report has been submitted to letter ballot of the committee, which consists of 39 members; 33 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

L. T. BISSEY,
Chairman.

R. H. MURRAY,
Secretary.

REPORT OF COMMITTEE D-4

ON

ON ROAD AND PAVING MATERIALS*

Committee D-4 on Road and Paving Materials held two meetings during the past year: in Atlantic City, N. J., on July 1, 1955, and in Buffalo, N. Y., on March 2, 1956.

The committee lost one member by death, namely, A. W. Dow, who was one of the original members. In recognition of Mr. Dow's many valuable contributions to the work of the committee and the loss which the members felt in his passing, an appropriate memorial resolution was adopted and this resolution is being entered on the permanent records of the committee and of the Society.

The officers elected for the ensuing term of two years are as follows:

Chairman, K. B. Woods.

First Vice-Chairman, A. B. Cornthwaite.

Second Vice-Chairman, Fred Hubbard.

Third Vice-Chairman, J. O. Izatt.

Secretary, J. M. Griffith.

NEW TENTATIVE

The committee recommends that the following method be accepted for publication as tentative as appended hereto:[1]

Method for:

Testing Graded Aggregate Road Mixes for Certain Water Soluble Chlorides Present as Admixes (jointly with Committee D-18).

REVISION OF TENTATIVE

The committee recommends that the Tentative Specifications for Preformed Expansion Joint Fillers for Concrete (Nonextruding and Resilient Types) (**D 544 – 52 T**)[2] be revised as follows and continued as tentative:

Section 3 (*b*).—Change to read as follows:

> The test specimen shall be compressed to 50 per cent of its thickness before test. The load shall be immediately released after application. At the end of 10 min after release of application, the specimen shall have recovered to at least 90 per cent of its thickness before tests for types I and II, and to at least 70 per cent for type III.
>
> In case of failure to comply with these requirements, the test specimen shall be given three applications of load sufficient to compress the material to 50 per cent of its thickness before test. The load shall be released immediately after each application. At the end of 1 hr after the third application the specimen shall have recovered as indicated above.

TENTATIVE REVISIONS OF STANDARDS

The committee recommends tentative revisions of the following standards as indicated:

Specifications for Preformed Expansion Joint Fillers for Concrete, Nonextruding and Resilient Types (D 544 – 49):[2]

Section 4 (*a*).—Change to read as follows:

> The test specimen shall be compressed to 50 per cent of its thickness before test. The load

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] The new tentative appears in the 1956 Supplement to Book of ASTM Standards, Part 3.

[2] 1955 Book of ASTM Standards, Part 3.

shall be immediately released after application. At the end of 10 min after release of application the specimen shall have recovered to at least 90 per cent of its thickness before test for types I, II, III, and IV, and to at least 70 per cent of its thickness before test for type V.

In case of failure to comply with the above requirements, the test specimen shall be given three applications of a load sufficient to compress material to 50 per cent of its thickness before test. The load shall be immediately released after each application. At the end of 1 hr after the third application the specimen shall have recovered as indicated above.

Specifications for Emulsified Asphalt (D 977 - 53):[2]

Section 1.—Delete the sixth paragraph pertaining to Type MS-3.

Section 2 (b).—Delete reference to and requirements for specific gravity.

Table I.—Delete reference to Type MS-3.

Specifications for Bituminous Mixing Plant Requirements (D 995 - 55):[2]

Section 2 (b).—Add the following to the end of this Section:

> "Separate tanks and pipe lines shall be provided when mixtures containing asphalt cement and liquid asphaltic materials are to be produced in the same mixer. When tar products or emulsified asphalt are to be used as the binding medium in bituminous mixtures, separate storage tanks as well as separate pipe lines shall be provided."

Section 2 (d).—Change the second sentence to read, "The drier shall be capable of drying and heating the aggregate to the temperature requirements set forth in the specifications without leaving any unburned oily residue on the aggregate when it is discharged from the drier."

Methods of Testing Preformed Expansion Joint Fillers for Concrete, Nonextruding and Resilient Types (D 545 - 49):[2]

Section 4 (d).—Change to read as follows:

> For the determination of the percentage of recovery, the test specimen shall be given a single application of a load sufficient to compress it to 50 per cent of its thickness before test. The load shall be applied without shock and at such a rate that the specimen will be compressed approximately 0.05 in. per min. The load shall be immediately released after application and permitted to recover 10 min, after which the thickness shall be measured. The load transferring apparatus and spherical bearing block shall be removed from the test specimen following the load application. The percentage of recovery shall be calculated as follows:
>
> $$\text{Recovery, per cent} = \frac{t'}{t} \times 100$$
>
> where:
>
> t = thickness of the specimen before test, and
> t' = thickness 10 min after completion of the application of load.
>
> In case the specimen fails to comply with requirements of the specification the test specimen shall be tested in accordance with Section 4 (*d*) of 1955 Book of ASTM Standards.

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption the following revision of the Method of Test for Specific Gravity of Compressed Bituminous Mixtures (**D 1188 - 53**)[2] and accordingly asks for the necessary nine tenths affirmative vote at the Annual meeting in order that the revision may be referred to letter ballot of the Society:

Section 4 (e).—Replace the reference to the Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (ASTM Designation: D 95) with a reference to a new Tentative Method for Determination of Moisture or Volatile Distillates in Bituminous Mixtures.[3]

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[3] This method will be presented to the Administrative Committee on Standards subsequent to the Annual Meeting; the revision of Method D 1188 can only become effective upon the approval of new method by the Standards Committee.

ADOPTION OF TENTATIVE REVISION AS STANDARD

The committee recommends that the tentative revision issued June, 1955 of the Standard Method of Test for Distillation of Tars and Tar Products (**D 20 - 55**)[2] be approved for reference to letter ballot of the Society for adoption as standard.

TENTATIVES CONTINUED WITHOUT REVISION

Committee D-4 recommends that the following specifications and methods be continued as tentative without revision. In all cases, subcommittees responsible for these tentatives consider that further work is required before recommending their adoption as standard:

Tentative Specifications for:

Asphalt Cement for Use in Pavement Construction (**D 946 - 47 T**),
Concrete Joint Sealer, Hot-Poured Elastic Type (**D 1190 - 52 T**),

Tentative Methods of Test for:

Loss on Heating of Oil and Asphaltic Compounds (**D 6 - 39 T**),
Moisture-Density Relations of Soils (**D 698 - 42 T**),
Soil-Bituminous Mixtures (**D 915 - 47 T**),
Shear Strength of Flexible Road Surfaces, Subgrades and Fills by the Burggraf Shear Apparatus (**D 916 - 47 T**),
Bitumen Content of Paving Mixtures by Centrifuge (**D 1097 - 54 T**),
Vacuum Distillation of Liquid and Semi-Solid Asphaltic Materials to Obtain a Residue of Specified Penetration (**D 1189 - 52 T**), and
Concrete Joint Sealers (**D 1191 - 52 T**).

EDITORIAL CHANGE

Committee D-4 recommends the following editorial change in the Method of Test for Soft Particles in Coarse Aggregates (**C 235 - 54**):

Title.—Change to read "Method of Test for Scratch Hardness of Coarse Aggregate Particles."

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee B-2, Physical Tests for Compressed Bituminous Mixtures (L. F. Rader, chairman) is continuing its activities in the preparation of methods of test for stability and flow measurements by the Marshall test apparatus, stabilometer and cohesiometer test measurements by the Hveem apparatus, and a method of compaction of bituminous specimens by the kneading compactor. The Marshall procedures have been submitted to the committee as information only. The subcommittee has also prepared, as information only, a proposed Hveem Method of Test for Resistance to Deformation and Cohesion of Bituminous Mixtures and a proposed method for preparing laboratory specimens for bituminous mixtures by the Kneading Compaction method.

The subcommittee has recommended the publication as tentative of the Method for Determination of Moisture or Volatile Distillates in Bituminous Mixtures, referred to previously in the report.[3] This method is identical with American Assn. of State Highway Officials Method T 110-42. The subcommittee considers this method to be superior to the Method of Test for Water in Petroleum Products and Other Bituminous Materials (D 95) for the determination of moisture in bituminous paving mixtures.

Subcommittee B-3, Distillation (J. W Donegan, chairman) is drafting a statement on precision for the Method of Test for Distillation of Cut-Back Asphaltic Products (D 402 - 49). The subcommittee is also working on proposed revisions of Method D 95 – 46.

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[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee B-5, Softening Point (D. F. Fink, chairman) is working on proposed revisions of the Method of Test for Softening Point by Ring and Ball Apparatus (E 28 – 51 T) with a view to making it a tentative revision of Method of Test for Softening Point of Bituminous Materials (D 36 – 26). A task group is examining Institute of Petroleum Method 58 on Softening Point in an effort to eliminate possible minor differences between the IP method and Method D 36.

Subcommittee B-6, Extraction and Recovery of Constituents from Bituminous Mixtures (R. R. Thurston, chairman) is investigating a possible source of error in penetration test results of recovered asphalts by Method D 762 – 49. A modification of the tube delivering CO_2 to the flask in the recovery portion of this method is being studied to determine if such a revision is required. The subcommittee also has under consideration a possible requirement for the use of filter paper, in addition to the felt ring, in Method of Test for Bitumen Content of Paving Mixtures by Centrifuge (D 1097 – 54 T).

Subcommittee B-12, Structural Properties of Mineral Aggregates (Stanton Walker, chairman) is considering incorporating in the Method of Test for Soft Particles in Coarse Aggregates (C 235–54) requirements for an apparatus to subject particles under test to a uniform load as well as other detailed changes. This work is being coordinated with Subcommittee III-e of Committee C-9 since Method C 235 is under the joint jurisdiction of Committees C-9 and D-4.

Subcommittee B-14, Specific Gravity of Fine and Coarse Aggregates (H. T. Williams, chairman) completed the arrangements for a Symposium on Specific Gravity of Bituminous Coated Aggregates"[5] to be presented at the 1956 Annual Meeting of the Society.

Subcommittee B-15, Sampling Road Materials (R. E. Britton, chairman) has under consideration several proposed revisions of Methods of Sampling Stone, Slag, Gravel, Sand, and Stone Block for Use as Highway Material (D 75) and Methods of Sampling Bituminous Paving Mixtures (D 979).

Subcommittee B-16, Setting Qualities of Bituminous Materials (A. B. Cornthwaite, chairman) has completed a cooperative test program with respect to the Rolling Ball test procedure for determining the rate of setting of bituminous materials. Further studies are being made on the effect of temperature on rate of flow, air currents in the curing ovens, angle of plate for Rolling Ball test and the use of different sizes of test balls.

Subcommittee B-17, Emulsion Tests (R. R. Thurston, chairman) is studying the repeatability and reproducibility of thirteen methods of test outlined in Method of Testing Emulsified Asphalts (D 244). Proposed methods of testing emulsified asphalts for stability are also being studied.

Subcommittee B-19, Accelerated Tests for Durability of Bituminous Materials (D. C. Taylor, chairman) through a task group is conducting cooperative tests on the thin film oven test as developed by the Bureau of Public Roads. The subcommittee is also making a study of the Shell Oil Co. micro thin film test.

Subcommittee B-24, Penetration Test (J. Y. Welborn, chairman) is considering a proposed revision of the Method of Test for Penetration of Bituminous

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[5] Issued as separate publication, *ASTM STP No. 191.*

Materials (D 5). Tentative specifications for a new needle to include closer control of the more critical features have been prepared, and a cooperative testing program will be carried out with these new needles.

Subcommittee B-25, Effect of Water on Compressed Bituminous Mixtures (C. A. Carpenter, chairman). has under consideration possible modifications of the Method of Test for Effect of Water on Cohesion of Compacted Bituminous Mixtures (D 1075 – 54), particularly with regard to the method of compaction.

Subcommittee B-26, Effect of Water on Bituminous Coated Aggregates (C. M. Hewett, chairman) is studying a variety of methods for the determination of the stripping resistance of aggregates used in bituminous paving mixtures.

Subcommittee C-4, Emulsified Asphalts (C. E. Proudley, chairman) has recommended the elimination of grade MS-3 and specific gravity of residue requirements from Specifications D 977 referred to previously in the report. The subcommittee now has under consideration a proposal to eliminate also grade MS-1 from the specifications.

Subcommittee C-8, Water-Bound Roads (R. R. Litehiser, chairman) is considering changes in the Specifications for Crushed Stone, Crushed Slag, and Gravel for Waterbound Macadam Bases and Surface Courses of Pavements (D 694 – 55). These suggested changes were largely editorial in nature but they will represent a major rearrangement of the various items included in the specifications. A revised draft of the specifications is therefore being submitted to all members of the subcommittee for consideration and comments.

Subcommittee C-12, Bituminous Surface Treatments (J. O. Izatt, chairman) is considering revisions of the Specifications for Crushed Stone, Crushed Slag, and Gravel for Single, Double, or Multiple Bituminous Surface Treatments (D 1139 – 55 T) with respect to definitions for crushed gravel, and limits for deleterious substances in the plus No. 4 portion of the aggregates.

Subcommittee C-13, Bituminous Paving Plant Operations (I. H. Boggs, chairman) prepared the tentative revisions of Specification D 995–55 referred to previously in the report.

Subcommittee D-1, Calcium Chloride and Sodium Chloride for Use as Road Materials (H. F. Clemmer, chairman) has under consideration proposed revisions of Specifications for Calcium Chloride (D 98 – 48) and Sodium Chloride (D 632–43).

Subcommittee D-2, Highway Traffic Markers (Paints, Insets and Strips) (N. G. Smith, chairman) is considering the development of more satisfactory specifications for permanent type traffic markers and proposed specifications for reflective materials for highway signs.

Subcommittee D-3, Expansion Joint Materials (H. F. Clemmer, chairman) has under consideration a number of items among which are the preparation of specifications for preformed expansion joint materials used in bridges and other highway structures; the preparation of specifications for jet fuel resistant joint sealer; the formulation of a method of test for extrusion of premolded joint filler under heat; a determination of the value of the hydrochloric acid test for cork premolded joint material; and the preparation of methods and specifications for determining the ability of joint materials to withstand handling at the construction site. The subcommittee has prepared the alternate test for determining the recovery of a premolded joint filler material after compression, referred to previously in the report, as tentative revisions of Specifications D 544 - 49 and D 544 - 52 T and Method D 545 - 49.

Joint Subcommittee of Committees D-4 and D-18, Tests and Specifications for Stabilized Soils (H. F. Clemmer, chairman) has prepared the proposed Tentative Method of Testing Graded Aggregate Road Mixes for Certain Water Soluble Chlorides Present as Admixes referred to previously in the report.

This report has been submitted to letter ballot of the committee, which consists of 166 members; 113 members returned their ballots, of whom 109 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

C. W. ALLEN,
Chairman.

J. M. GRIFFITH,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-4 presented to the Society through the Administrative Committee on Standards the recommendation that the Standard Specifications for Calcium Chloride (D 98 - 48) be revised and reverted to tentative status. This recommendation was accepted by the Standards Committee on November 15, 1956, and the tentative specifications appear in the 1956 Supplement to Book of ASTM Standards, Part 3.

REPORT OF COMMITTEE D-5

ON

COAL AND COKE*

Committee D-5 on Coal and Coke held one meeting during the year: in Atlantic City, N. J., on June 28, 1955; in addition, subcommittee meetings were held in Columbus, Ohio, on October 18–21, 1955, and in Buffalo, N. Y., on February 27–29, 1956.

During the year two members were added to the committee, resulting in a total voting membership of 61; of whom 16 are classified as producers, 22 as consumers, and 23 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, O. N. Rees.
Vice Chairman, R. L. Coryell.
Secretary, C. C. Russell.

NEW TENTATIVE

The committee recommends that the Method of Test for Equilibrium Moisture of Coal at 96–97 per cent Relative Humidity and 30 C be accepted for publication as tentative as appended hereto.[1]

TENTATIVE REVISION OF STANDARD

The committee recommends the following tentative revision of the Standard Definitions of Terms Relating to Coal and Coke (**D 121 – 30**):[2]

Add the following new definition:

Equilibrium Moisture of Coal, is the moisture content retained at equilibrium in an atmosphere over a saturated solution of potassium sulfate at 30 C, and 96 to 97 per cent relative humidity. When the sample, before such equilibration, contains total moisture at or above the equilibrium moisture, the equilibrium moisture may be considered as equivalent to *inherent* or *bed* moisture; and any excess may be considered as *extraneous* moisture.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Nomenclature and Definitions (C. H. Sawyer, chairman) prepared the tentative revision of the Standard Definitions of Terms Relating to Coal and Coke (D 121 – 30) referred to earlier in the report, consisting of a definition for Equilibrium Moisture of Coal at 96 to 97 per cent relative humidity and 30 C. This new definition is required for use in conjunction with the new Method of Test for Equilibrium Moisture of Coal.

Subcommittee VII on Pulverizing Characteristics of Coal (H. F. Yancey, chairman) is drafting a revision of the Standard Method of Test for Grindability of Coal by the Hardgrove Machine Method (D 409 – 51) to provide more rigorous procedures for sample preparation and sieving techniques. Investigation is being made of the possibility of increasing the size range of the laboratory sample used in the Hardgrove grindability machine.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] The new tentative appears in the 1956 Supplement to Book of ASTM Standards, Part 5.

[2] 1955 Book of ASTM Standards, Part 5.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee XV on Plasticity and Swelling of Coal (Michael Perch, chairman) is seeking data through ISO/TC 27 regarding the repeatability and reproducibility of the Audibert-Arnu dilatometer, as well as a correlation of the results of the test with the coking properties of various ranks of coals.

The subcommittee is conducting a new and extensive series of tests to determine operating variables of the Gieseler plastometer. A series of tests is to be made on completely standardized apparatus to determine repeatability and reproducibility of the method, all results to be analyzed statistically in accordance with a previously approved design of experiment.

The subcommittee is considering recommending the tentative revision of the Standard Method of Test for Free Swelling Index of Coal (D 720 - 46) for adoption as standard.

Subcommittee XX on Sampling and Fineness Test of Pulverized Coal (E. L. Rogers, chairman) is working on the second draft of a proposed revision of the Standard Method of Sampling and Fineness Test of Powdered Coal (D 197 - 30), the object of the revision being to include the latest techniques and to specify the testing procedures in greater detail.

Subcommittee XXI on Methods of Analysis (O. W. Rees, chairman) prepared the new Tentative Method of Test for Equilibrium Moisture of Coal at 96 to 97 per cent Relative Humidity and 30 C, referred to earlier in the report. This method affords a means for estimating the bed moisture of coal and also for estimating the surface or extraneous moisture of wet coal. The tentative revision of the Standard Method of Laboratory Sampling and Analysis of Coal and Coke (D 271 - 48) has elicited several suggestions regarding the revised carbon and hydrogen methods.

Subcommittee XXIII on Sampling (W. M. Bertholf, chairman): Section A on Coal Sampling is conducting a series of experiments to obtain variance *versus* increment weight curves on a number of coals of the United States and Canada, which data will provide sampling characteristics of coals with known histories that can be used as the basis for specifying sampling requirements for various classes of coals.

As a result of the conclusions reached at the Coal Symposium held in June, 1954, Section C on Sample Preparation is experimenting with various types of sample splitters in an attempt to reduce the variances at various stages of sample preparation. Investigation is also being made of the possibility of reducing sample preparation to a minimum of two stages.

This report has been submitted to letter ballot of the committee, which consists of 61 voting members; 58 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. W. ANDERSON,
Chairman.

O. P. BRYSCH,
Secretary.

REPORT OF COMMITTEE D-6

ON

PAPER AND PAPER PRODUCTS*

Committee D-6 on Paper and Paper Products held two meetings during the year: on June 30, 1955, in Atlantic City, N. J., and on February 24, 1956, in New York City. The Advisory and other subcommittees held meetings in conjunction with the main committee.

The committee consists of 95 members, of whom 81 are voting members; 36 are classified as producers, 26 as consumers, and 33 as general interest members.

A method of maintaining closer cooperation with the Technical Association of the Pulp and Paper Industry has been implemented through the establishment of joint task groups for individual test methods. When details of a method are worked out by the task group, approval will proceed along previously established procedures by each organization. This procedure should eliminate duplication of effort and keep the standards of the two societies in closer agreement.

The officers elected for the ensuing term of two years are as follows:

Chairman, R. H. Carter.
Vice-chairman, H. A. Birdsall.
Secretary, R. E. Green.

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Methods of Testing Analytical Filter Papers (**D 981 – 51 T**)[1] be approved for reference to letter ballot of the Society for adoption as standard with editorial revision of Section 5.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following eight tentative methods, which have stood for two years without revision, be continued as tentative:

Tentative Method of Test for:

Puncture and Stiffness of Paperboard, Corrugated and Solid Fiberboard (**D 781 – 44 T**),
Stretch of Paper and Paper Products Under Tension (**D 987 – 48 T**),
Water Vapor Permeability of Paper and Paperboard (**D 988 – 51 T**),
Chloride Content of Paper and Paper Products (**D 1161 – 51 T**),
Pinholes in Glassine and Other Greaseproof Papers (**D 1221 – 52 T**),
Contrast Gloss of Paper at 57.5 deg (**D 1222 – 52 T**),
Specular Gloss of Paper at 75 deg (**D 1223 – 52 T**), and
Dimensional Changes of Paper with Changes in Moisture Conditions (**D 1270 – 53 T**).

EDITORIAL CHANGES

The committee recommends that the following standard be editorially revised as indicated:

Standard Method of Test for Pentosans in Paper (D 688 – 44):[1]

Section 2(b).—In the sixth line, after the word "long," add the word "spiral."

Section 7.—Change to read as follows:

7. Calculate the pentosan content of the paper as follows:

$$\text{Pentosans, per cent} = \frac{7.5N(v_2 - v_1)}{W} - 1.0$$

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 18–22, 1956.

[1] 1955 Book of ASTM Standards, Part 7.

where:

N = normality of thiosulfate solution,
v_1 = volume of thiosulfate solution used in test,
v_2 = volume of thiosulfate solution used in blank, and
W = weight of specimen after correcting for moisture.

NOTE.—The factor 7.5 is the product of

$$\frac{100 \times 0.048}{0.727 \times 0.88}$$

where: 0.048 is the weight of furfural in grams corresponding to 1 ml of N thiosulfate solution; 0.727 is the theoretical conversion factor of pentosans to furfural; and 0.88 is a factor to compensate for the incomplete conversion of pentosans to furfural. The correction factor of 1.0 is applied to compensate for the hydroxymethylfurfural arising from the cellulose.

The factor 0.88 is based upon the observation that the yield of furfural from xylose is 88 per cent of the theoretical in this method of distillation. The corresponding value for arabinose is 74 per cent, and a furfural yield of 80 per cent is frequently taken as an approximate average in calculating furfural yields to pentosans. However, since xylan constitutes most if not all of the pentosans in wood pulp, it appears preferable to use the factor 0.88. If both araban and xylan are present, the above equation becomes:

$$\text{Pentosans, per cent} = \frac{8.25N(v_2 - v_1)}{W} - 1.1$$

This equation with the omission of the 1.1 per cent correction, gives values which are comparable with those obtained by using Krober's tables, in which the conversion of pentosans to furfural is also arbitrarily taken as 80 per cent.

The factors 0.80 and 0.88 which are applied to compensate for the incomplete conversion of pentosans to furfural are somewhat arbitrary. If the analyst desires values in which these factors do not appear, and in which the furfural actually obtained is converted to equivalent pentosans, a minimum pentosan content may be calculated by the equation:

$$\text{Pentosans, per cent} = \frac{6.80\,N\,(v_2 - v_1)}{W} - 0.9$$

where:

$$6.80 = \frac{1.03 \times 0.0480 \times 100}{0.727}$$

The factor 1.03 is the correction necessary to account for the destruction of furfural during the distillation.

W = weight of specimen after correcting for moisture, ash, and any filling and sizing materials present, and
0.9 = constant to correct for hydromethyl furfural.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Sampling and Conditioning (R. H. Lace, chairman) is continuing its work on a revision of the Method of Sampling Paper and Paper Products (D 585 – 42).

Subcommittee II on Chemical Test Methods (E. G. Ham, chairman) is carrying out investigations which may lead to proposed revisions in the Method of Test for Starch in Paper (D 591 – 42), Method of Qualitative Examination of Mineral Filler and Mineral Coating of Paper (D 686 – 48), Method for Quantitative Determination of Coating on Mineral-Coated Paper (D 687 – 44), Method of Test for Reducible Sulfur in Paper (D 984 – 50), Method of Test for Titanium Dioxide in Paper (D 921 – 49), and Method of Test for Chloride Content of Paper and Paper Products (D 1161 – 51 T).

Subcommittee III on Physical Test Methods for Paper (L. Price, chairman) recommended the adoption as standard of the Tentative Methods of Testing Analytical Filter Paper (D 981 – 51 T).

Subcommittee IV on Physical Test Methods for Container Board (W. B. Lincoln, Jr., chairman) is continuing its work on a revision of the Method of Test for Puncture and Stiffness of Paperboard, Corrugated and Solid Fiberboard (D 781 – 44 T) and is working toward a proposed revision of the Method for Flat Crush of Corrugated Paperboard (D 1225 - 54). Work is also under way on a method for evaluating corrugating mediums.

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[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee VI—Editorial (L. S. Reid, chairman) is continuing its work of editing and preparing sections on significance of test for all of the methods.

This report has been submitted to letter ballot of the committee, which consists of 81 voting members; 46 members returned their ballots, of whom 41 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. R. WILLETS,
Chairman.

R. H. CARTER,
Secretary.

REPORT OF COMMITTEE D-7

ON

WOOD*

Committee D-7 on Wood met in Chicago, Ill., on March 12 and 13, 1956. The following subcommittee meetings preceded the main committee meeting: Subcommittee VII on Wood Poles and Cross Arms, Subcommittee XI on Moisture Content, Subcommittee XIII on Durability and Exposure, and Subcommittee XV on Structural Fiberboards.

Eleven applicants were elected to membership on the committee; there were six resignations and one death. One new subcommittee chairman was appointed during the year—Wayne E. Moore, Subcommittee XIV on Methods of Chemical Analysis.

The officers elected for the ensuing term of two years are as follows:

Chairman, L. J. Markwardt.
Secretary, L. W. Smith.

NEW TENTATIVE

The committee recommends that the Method of Testing Wood Preservatives by Laboratory Soil-Block Cultures be accepted for publication as tentative as appended hereto.[1]

REVISION OF TENTATIVES

The committee recommends that the Tentative Method of Test for Evaluating the Properties of Building Fiberboards (**D 1037 – 52 T**)[2] be revised to include the methods for nail-head pull through, block shear, and glue-line shear, as appended hereto,[1] and continued as tentative.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Specifications for:

Copperized Chromated Zinc Chloride (**D 1271 – 53 T**),[2]
Pentachlorophenol (**D 1272 – 53 T**),[2]
Ammoniacal Copper Arsenite (**D 1325 – 54 T**),[2]

Methods for Chemical Analysis of:

Copperized Chromated Zinc Chloride (**D 1273 – 53 T**),[2]
Pentachlorophenol (**D 1274 – 53 T**),[2]
Ammoniacal Copper Arsenite (**D 1326 – 54 T**),[2]

Methods of Test for:

Ash in Wood (**D 1102 – 50 T**),[2]
Holocellulose in Wood (**D 1104 – 50 T**),[2]
Lignin in Wood (**D 1106 – 50 T**)[2]
Alcohol-Benzene Solubility of Wood (**D 1107 – 50 T**),[2]
Ether Solubility of Wood (**D 1108 – 50 T**),[2]
One Per Cent Caustic Soda Solubility of Wood (**D 1109 – 50 T**),
Water Solubility of Wood (**D 1110 – 50 T**), and

Method for Preparation of:

Extractive-free Wood (**D 1105 – 50 T**).

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following tentatives be continued in their present status pending further study:

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 18–22, 1956.

[1] The new tentative appears in the 1956 Supplement to Book of ASTM Standards, Part 4.

[2] 1955 Book of ASTM Standards, Part 4.

Tentative Specifications for:

Modified Wood (**D 1324 - 54 T**), and

Tentative Methods of:

Establishing Structural Grades of Lumber (**D 245 - 49 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Specifications for Timber (L. W. Wood, chairman) continues to study possible revisions of the Tentative Methods for Establishing Structural Grades of Lumber (D 245 - 49 T).

Subcommittee II on Laminated Timber (Frank J. Hanrahan, chairman).—In view of the developments in testing practice with respect to glue joints, the subcommittee is working on a complete revision of the Standard Method of Test for Integrity of Glue Joints in Laminated Wood Products for Exterior Service (D 1101 - 53).

Subcommittee VI on Timber Preservatives (D. L. Davies, chairman).—Subcommittee 19 on Glass Laboratory Apparatus of ASTM Committee E-1 suggested several changes in the design of the tar-acid separatory funnels illustrated and specified in the Test for Water in Creosote (D 370 - 33) and Test for Tar Acids in Creosote and Creosote-Coal Tar Solutions (D 453 - 41). The suggestions are being held for further consideration.

Subcommittee VII on Wood Poles and Cross Arms (R. P. A. Johnson, chairman).—The ASTM Wood Pole Research Program has occupied most of the subcommittee's attention. The tests on lodgepole pine poles have resulted in lower stress values than had been anticipated, which has raised the question as to whether samples of lower than average specific gravity had been used and whether some lodgepole pine producing areas had been sampled adequately. A check on the age and specific gravity of poles now on the market in pole yards is being made. Regardless of previous sampling, the lodgepole pine poles for preservative treatment will be selected to match as nearly as possible the untreated poles in accordance with the original working plan.

At present, all untreated poles included in the research program have been tested and the interim reports on Douglas-fir, southern pine, and western larch poles have been printed and distributed. Some treated poles are being delivered and tests are starting. The work will not be finished for another year and a half, and no active reconsideration of pole stresses by the ASA Sectional Committee O 5 can be undertaken until the ASTM data are analyzed and reported.

In order to facilitate the orderly progress of the work, the Ways and Means Advisory Group suggested that the program be established on a three- rather than a two-year basis. Due to increased costs and additional work included since the work was planned several years ago, the total estimated cost is now $254,000. To date about $160,000 has been collected. Contributions are being invited to underwrite the completion of the work.

Subcommittee XI on Moisture Content of Timber (R. C. Rietz, chairman) held an organization meeting on March 12, 1956. Since the oven-dry method is the only procedure in ASTM standards for determining the moisture content of timber, several additional methods will be studied during the coming year.

Subcommittee XIII on Durability and Exposure (R. M. Lindgren, chairman).—

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

The principal effort during the year was in the development of the proposed Method of Testing Wood Preservatives by Laboratory Soil-Block Cultures referred to previously in this report. The usual method of evaluating wood preservatives is by exposure of treated specimens to contact with the ground in outdoor sites. The new method, based on developments in progress for some 15 years, provides a soil-block technique as a laboratory procedure for testing wood preservatives. Initial trials of the soil-block method were made by the Bell Telephone Laboratories. Similar developmental work has been in progress at the Forest Products Laboratory for the past 10 years. Research on the method also has been carried out at several other governmental, state, and private laboratories in the United States and Canada. Such developmental work during the past 5 years has been accompanied by actual use of the method in supplying information on a number of problems. The method is being used as a laboratory tool in Australia, New Zealand, Sweden, Indonesia, and several other countries, as well as in the United States and Canada.

The subcommittee expects to consider an accelerated laboratory test method for evaluating natural decay resistance of wood, and consideration may be given to accelerated field testing methods for evaluating both preservatives and natural decay resistance of wood. Available for consideration is an American Wood Preservers Association procedure for accelerated field testing of preservatives, involving the use of $\frac{3}{4}$-in. and 2- by 4-in. stakes.

Subcommittee XIV on Methods of Chemical Analysis (Wayne E. Moore, chairman) has reviewed the standards with which it is concerned and recommends that eight tentatives be adopted as standard.

Subcommittee XV on Structural Fiberboards (W. C. Lewis, chairman) has investigated preservative treatments to protect fiberboards against termite attack and decay. It is expected that a draft of a proposed standard nomenclature will be ready for correlation with Subcommittee X during the coming year. Three additional test procedures for nail-head pull through, block shear, and glue-line shear have been developed which are recommended for incorporation in the Tentative Methods of Evaluating the Properties of Building Fiber-Boards (D 1037 – 52 T).

This report has been submitted to letter ballot of the committee, which consists of 94 voting members: 53 members returned their ballots, of whom 52 have voted affirmatively and 0 negatively.

Respectfully submitted in behalf of the committee,

L. J. MARKWARDT,
Chairman.

L. W. Smith,
Secretary.

REPORT OF COMMITTEE D-8

ON

BITUMINOUS WATERPROOFING AND ROOFING MATERIALS*

Committee D-8 on Bituminous Waterproofing and Roofing Materials held two meetings during the year: in Atlantic City, N. J., on June 29, 1955, and in Buffalo, N. Y., on February 29, 1956.

The committee suffered loss through the death of J. H. Holden of the Pittsburgh Testing Laboratory.

The committee also lost through retirement, E. O. Rhodes of the Koppers Co., a longtime member of the committee.

The membership, consisting of 77 voting members, is composed of 49 producers, 11 consumers, and 17 general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, H. R. Snoke.
Vice Chairman, M. R. Beasley.
Secretary, G. W. Robbins.

On June 29, 1955, the committee held its 50th Anniversary Luncheon. The speakers were H. R. Snoke, chairman, N. L. Mochel, President, and R. J. Painter, Executive Secretary, respectively, of the Society.

All of the standards and tentatives for which the committee is responsible have been reviewed and recommendations concerning them are set forth in this report.

REVISION OF TENTATIVE

The committee recommends that the following tentative specifications be revised as indicated and continued as tentative:

Tentative Specification for Woven Burlap Fabrics Saturated with Bituminous Substances for Use in Waterproofing (D 1327 - 54 T):[1]

Section 1.—In the fourth line, change "coal-tar pitch" to read "refined coal-tar."

Section 2 (a).—In the first line, change "For the construction of" to read "For the plying cement employed in the construction of."

Section 2 (b).—In the first line, change "For the construction of" to read "For the plying cement employed in the construction of."

Section 3.—In the fourth line, change "coal-tar pitch" to read "refined coal-tar."

Table I.—For moisture content based on net weight per cent, change maximum of "7" to "4."

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Specification for Asphalt Roll Roofing Surfaced with Mineral Granules (**D 249 - 54 T**)[1] be approved for reference to letter ballot of the Society for adoption as standard without revision.

ADOPTION OF TENTATIVE REVISIONS AS STANDARD

The committee recommends that tentative revisions of the following standards be approved for reference to letter ballot of the Society for adoption as standard:

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* Pesented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] 1955 Book of ASTM Standards, Part 3.

Standard Specification for Asphalt-Saturated Felt for Use in Waterproofing and in Constructing Built-Up Roofs (**D 226 - 47**):[1] Tentative Revision of Section 9 issued June, 1954,

Standard Specification for Coal-Tar Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs (**D 227 - 47**):[1] Tentative Revision of Section 8 issued June, 1954, and

Standard Specification for Asphalt-Saturated Asbestos Felts for Use in Waterproofing and in Constructing Built-Up Roofs (**D 250 - 47**):[1] Tentative Revision of Section 9 issued June, 1954.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following specifications and methods be continued as tentative without revision, since they are being reviewed for possible revision:

Tentative Specification for:

Asphalt-Base Emulsions for Use as Protective Coatings for Built-Up Roofs (**D 1227 - 52 T**),

Asphalt Insulating Siding Surfaced with Mineral Granules (**D 1226 - 52 T**).

Tentative Method of:

Testing Asphalt-Base Emulsions for Use as Protective Coatings for Built-Up Roofs (**D 1167 - 51 T**),

Test for Staining Properties of Asphalts (Modified Pressure Method) (**D 1328 - 54 T**), and

Testing Asphalt Insulating Siding Surfaced with Mineral Granules (**D 1228 - 52 T**).

Tentative Recommended Practice for:

Accelerated Weathering Test of Bituminous Materials (**D 529 - 39 T**).

TENTATIVE REVISION CONTINUED WITHOUT CHANGE

The committee recommends that the tentative revision of Standard Specifications for Asphalt Shingles Surfaced with Mineral Granules (**D 225 - 54**)[1] be continued without change.

EDITORIAL CHANGES

The committee recommends the following editorial changes in the Standard Specification for Asphalt Roll Roofing Surfaced with Powdered Talc or Mica (**D 224 - 55**):[1]

Section 6 (*b*).—Change to read: "The coating and surfacing shall be applied uniformly on each side and up to the edges of the sheet."

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

This report has been submitted to letter ballot of the committee, which consists of 77 members; 44 members returned their ballots, of whom 42 have voted affirmatively and 0 negatively.

Respectfully submitted in behalf of the committee,

H. R. SNOKE,
Chairman.

G. W. ROBBINS,
Secretary.

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[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-8 presented to the Society through the Administrative Committee on Standards the recommendation that the Standard Methods of Sampling and Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing and Roofing (D 146 - 47) be revised and reverted to tentative status. This recommendation was accepted by the Standards Committee on September 5, 1956, and the tentative methods appear in the 1956 Supplement to Book of ASTM Standards, Part 3.

REPORT OF COMMITTEE D-9 ON ELECTRICAL INSULATING MATERIALS[*]

Committee D-9 held three meetings during the year: in Atlantic City, N. J., during the Annual Meeting of the Society; in Cincinnati, Ohio, on November 17, 18, and 19, 1955; and in Philadelphia, Pa., on February 13, 14, and 15, 1956.

At each of these meetings, action was taken on new tentatives, revision of existing tentatives, and changes in standards under jurisdiction of the committee. Also, progress was made on other projects under study by the subcommittees, as presented later in this report.

On February 15, 1956, the recently authorized Subcommittee X on Magnet Wire Insulation (K. N. Mathes, chairman) held its first meeting. A summary of its proposed activities is included later in this report.

Committee D-9 has unanimously elected W. A. Zinzow an Honorary Member of the committee in appreciation of his long term of service and his many contributions to the committee's work.

The committee submits for publication the paper by H. R. Meahl entitled Dissipation Factor and Dielectric Constant *versus* Frequency for Four Popular Materials, as appended to this report.[1]

The officers elected for the ensuing term of two years are as follows:

Chairman, H. K. Graves.
Vice-Chairman, F. M. Clark.
Recording Secretary, J. E. Gibbons.
Membership Secretary, J. R. Perkins.

Recommendations Accepted by the Administrative Committee on Standards

Subsequent to the 1955 Annual Meeting, the committee presented the following recommendations to the Society through the Administrative Committee on Standards:

Tentative Method of Test for:

Dielectric Proof-Voltage Testing of Thin Solid Electrical Insulating Materials (**D 1389 - 56 T**).

Revision of Tentative Methods of:

Testing Pasted Mica used in Electrical Insulation (**D 352 - 55 T**). (Comprises changes in Stability Under Heat and Pressure and in Mica or Binder Content.)

Test for High-Voltage, Low Current Arc Resistance of Solid Electrical Insulating Materials (**D 495 - 48 T**). (Comprises transfer of "Volts after tracking" sections to an appendix with appropriate editorial changes.)

Test for Silicone Insulating Varnishes (**D 1346 - 54 T**). (Comprises addition of Time of Drying instructions.)

Revision and Reversion to Tentative of Standard Method for:

Testing Pasted Mica Used in Electrical Insulation (**D 352 - 52**).

The revision of Standard Method D 352 - 52 and reversion to tentative, was accepted by the Standards Committee on September 15, 1955, and the additional revisions of Method D 352 - 55 T were accepted on April 2, 1956. New tentative method D 1389 - 56 T was accepted on January 27, 1956. The revisions of Methods D 495 - 48 T and of

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[*] Presented at the Fifty-ninth Annual Meeting of the Society, June 18–22, 1956.

[1] See p. 312.

D 1346 - 54 T were accepted on April 2, 1956.

The new and revised tentatives appear in the 1956 Compilation of ASTM Standards on Electrical Insulating Materials.

PROPOSED SPECIFICATION TO BE PUBLISHED AS INFORMATION

The committee recommends that the Proposed Specification for Electrical Insulating Paper Sulfate (Kraft) Tissue for Capacitor Dielectric be accepted for publication as information only as appended hereto.[2]

TENTATIVE REVISION OF STANDARD

The committee, jointly with Committee D-20 on Plastics, recommends tentative revisions as follows of the Standard Methods of Conditioning Plastics and Electrical Insulating Materials (**D 618 - 54**):[3]

Section 3.—Change the title of Paragraph (*d*) to read "Standard Test Temperatures other than Standard Laboratory Temperature."

Change list of temperatures and tolerances to read as follows:

Test Temperatures	Tolerance plus or minus
−70 C (−94 F)	2.0 C (3.6 F)
−55 C (−67 F)	2.0 C (3.6 F)
−40 C (−40 F)	2.0 C (3.6 F)
−25 C (−13 F)	2.0 C (3.6 F)
0 C (32 F)	2.0 C (3.6 F)
35 C (95 F)	1.0 C (1.8 F)
50 C (122 F)	2.0 C (3.6 F)
70 C (158 F)	2.0 C (3.6 F)
90 C (194 F)	2.0 C (3.6 F)
105 C (221 F)	2.0 C (3.6 F)
120 C (248 F)	2.0 C (3.6 F)
130 C (266 F)	2.0 C (3.6 F)
155 C (311 F)	2.0 C (3.6 F)
180 C (356 F)	2.0 C (3.6 F)
200 C (392 F)	3.0 C (5.4 F)
250 C (482 F)	3.0 C (5.4 F)
300 C (572 F)	3.0 C (5.4 F)

Section 4.—Add a second sentence to Note 1 to read as follows:

> Note that "Procedure A" of Methods D 618 differs from "Condition A" of ASTM Methods D 709 and of the Armed Forces Specifications MIL-P designation in that "Condition A" means "As received, no special conditioning."

Change the tolerance on humidity in Paragraph (*b*) from "±3C(±5.4F)" to "±2C±(3.6F)" to conform to the temperature table.

Add a third sentence to Note 3 to read as follows:

> Note that Procedure B of Methods D 618 is the same as Condition E-48/50 of ASTM Specifications D 709 and of the Armed Forces Specifications MIL-P designation.

Change the temperature tolerance in Paragraph (*c*) to ± 1C (1.8 F).

REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommends that the Standard Methods of Testing Solid Filling and Treating Compounds Used for Electrical Insulation (**D 176 - 44**)[3] be revised as appended hereto[4] and reverted to tentative status.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without change:

Tentative Methods of Testing:

Askarels (**D 901 - 55 T**),[3]

Tentative Specification for:

Enclosures and Servicing Units for Tests Above and Below Room Temperature (**D 1197 - 54 T**).[3] (Jointly with Committee D-20.)

ADOPTION OF TENTATIVE REVISIONS AS STANDARD

The committee recommends that the tentative revisions of the following standards be approved for reference to letter ballot of the Society for adoption as standard:

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[2] Published in the Compilation of ASTM Standards on Electrical Insulating Materials, 1957.

[3] 1955 Book of ASTM Standards, Part 6.

[4] The new tentative appears in the 1956 Supplement to Book of ASTM Standards, Part 6.

Methods of:

Testing Laminated Tubes Used for Electrical Insulation (**D 348 – 52**),[3]
Testing Laminated Round Rods Used for Electrical Insulation (**D 349 – 52**),[3]
Sampling Electrical Insulating Oils (**D 923 – 49**).[3]

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions of the Standard Methods of Testing Varnished Glass Fabrics and Varnished Glass Fabric Tapes used for Electrical Insulation (**D 902 – 53**)[3] as appended hereto,[5] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society.

EDITORIAL CHANGES

The committee recommends editorial changes in the following methods of test as indicated:

Tentative Methods of Test for Dielectric Breakdown Voltage and Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies, (D 149 – 55 T):[3]

Appendix.—Add as an appendix the section entitled "Significance of the Dielectric Strength Test" published as information in the Compilation of ASTM Standards on Electrical Insulating Materials, February, 1955, pp. 587–592.

Standard Methods of Testing Varnishes Used for Electrical Insulation (D 115 – 55):[3]

Section 29(b).—Change "0.0025 in." to read "0.0078 in." to conform to stated thickness of kraft paper in Section 20(*b*).

Tentative Methods of Testing Electrical Insulating Oils (D 117 – 54 T):[3]

Section 15.—Change the last sentence to read: "Under certain conditions, when the interfacial tension drops below a certain value, it may be an indication that sludge precipitation in the transformer has started or is imminent."

Standard Method of Test for Dielectric Strength of Insulating Oil of Petroleum Origin (D 877 – 49):[3]

Section 1.—In line 3, before "transformers," add the word "cable."

Section 6(a).—After the heading "Preparation of Sample," add a reference to a new footnote 3 which should read as follows:

[3] In the case of viscous oils having viscosities ranging between 60 sec and 100 sec SSU at 210 F, it is impracticable to handle these heavy oils according to the procedure in Section 6(a). In testing high-viscosity oils in the above range, the sample should be allowed to stand until it reaches room temperature, which should not be less than 20 C (68 F). The sample container should not be tilted, or swirled as prescribed in Section 6(*a*), and it should be opened only when the sample is ready for testing. The size of the sample should be selected so that the final cup filling will contain the normal drainings from the container.

Section 8.—Change Paragraph (1) to read: "Procedure used, whether Paragraph (*a*) or (*b*) of Section 7, or Section 6(*a*) or footnote under 6(*a*)."

Standard Method of Test for Inorganic Chlorides and Sulfates in Insulating Oils (D 878 – 49):[3]

Section 4(a).—Add a reference to a new Footnote 3 which should read as follows:

[3] In the case of viscous oils, the separated water may not be clear. If such is the case, the water layer should be filtered through a 10-cm diameter qualitative filter paper into a 250-ml beaker. The filtrate is then used for the tests. As a check on the filter paper, 75 ml of hot distilled water, passed through the paper prior to

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[5] The revised standard appears in the 1956 Supplement to the Book of ASTM Standards, Part 6.

filtering the separated water, should give negative tests for chlorides and sulfates.

The tentative methods and specifications under the jurisdiction of Committee D-9 which are not the subject of recommendations in this report are being continued as tentative for further study.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[6]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Insulating Varnishes, Paints and Lacquers (W. E. Harvey, chairman) prepared the revision of the Tentative Method of Testing Silicone Insulating Varnishes (D 1346 - 54 T) by addition of a test for time of drying, as noted under Recommendations Accepted by the Administrative Committee on Standards. The recommended addition is based on the time-of-drying procedure in the Tentative Methods of Testing Insulating Varnishes (D 115); but is designed particularly for use with the silicone varnishes.

Under study is the need for including in Methods D 1346 - 54 T sections on drainage test, oil resistance, heat shock, coatability, and cure time.

In Methods D 115 - 55, the subcommittee recommends a minor editorial change in Section 29(*b*), as noted earlier in this report.

Other work in progress comprises continuance of studies in Section C on set time of laminating varnishes; on heat stability in Section G; and on means of supplementing or replacing the MacMichael Viscosimeter for measuring viscosity of varnish in Section J.

The name of Section D has been changed from "Silicone Varnishes" to "Heat-Resistant Varnishes."

Mr. H. C. Gunst succeeds Mr. J. B. Hyman as Chairman of Section J on Physical Tests.

Subcommittee III on Plates, Tubes, Rods and Molded Materials (E. A. Russell, chairman) recommends adoption as standard of Tentative Revisions of Methods D 348 - 52 and D 349 - 52. Both methods have had attached to them since June, 1954, tentative revisions of some sections under the heading Editorial Note.

Work in progress comprises a proposed method of measuring insulation resistance to be included in Method of Testing Sheet and Plate Material Used for Electrical Insulation (D 229 - 49); a study of electrodes for measuring surface resistivity of CSI XXXP Laminate; a proposed revised specification for the same material; a comparison of ASTM and International Electrotechnical Commission methods for measuring dielectric strength parallel to laminations of laminated materials; and a study of a proposed test method for heat resistance and thermal stability of rigid electrical insulation.

Subcommittee IV on Liquid Insulation (F. M. Clark, chairman).—The subcommittee recommends that Method D 901 - 53 T and tentative revision of Standard Method D 923 - 49 be adopted as standard.

The subcommittee also recommends adoption of editorial changes in Methods D 117 - 54 T, D 877 - 49, and D 878 - 49, as noted earlier in this report. The purpose of the change in Methods D 117 - 54 T is to clarify the relation between interfacial tension and sludge formation in oxidized transformer oil. The purpose of the changes in Methods D 877 - 49 and D 878 - 49 is to extend the scope of the two methods to viscous cable oils.

Activities of the subcommittee are carried on by twenty-five sections, covering a wide variety of projects. A partial list includes oxidation stability of inhibited and uninhibited transformer oil; methods for analysis for traces of metals in insulating oils; a survey of tests applicable to cable impregnating oils; measurement of inhibitor content of inhibited oils; study of cells for measuring power factor and resistivity of oils, and of means of checking accuracy of power factor bridges by use of an RC test standard; improvement of apparatus for measuring gas solubility in insulating oils; study of tests for measuring gas absorption or evolution in oils in a corona discharge; and application of statistical methods for evaluation of data.

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[6] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Cooperation with other committees is effected by representation on American Institute of Electrical Engineers committees on Instruments and Measurements; Transformers; and Insulated Conductors. Other cooperative relations are with the AIEE Committee on Dielectrics, the International Congress for High-Voltage Research, and the International Electrotechnical Commission.

Membership classification has been revised to promote more effective work by the subcommittee. The classification comprises active members, who participate in section programs; inactive members, who are interested in work of the subcommittee but are not able to take active part; and consulting members, who furnish specialized but temporary help on some problems.

At the meeting of the subcommittee in Philadelphia on February 15, 1956, a Survey Section was organized to review the need and desirability of continuing present sections and of forming new sections.

Subcommittee VI on Solid Filling and Treating Compounds (W. R. Dohan, chairman) prepared a complete revision of the Standard Methods of Testing Solid Filling and Treating Compounds Used for Electrical Insulation (D 176 – 44) and recommends that the method be reverted to tentative status, as noted above.

Subcommittee VII on Insulating Fabrics (H. K. Graves, chairman) has completed a revision of the Standard Methods of Testing Varnished Glass Fabrics and Varnished Glass Fabric Tapes Used for Electrical Insulation (D 902 – 53) and recommends its immediate adoption as standard, as indicated above.

First drafts of specifications for yellow bias-cut varnished cloth and for black and yellow straight-cut varnished cloths have been circulated and discussed.

Methods for testing silicone rubber glass fiber, polytetrafluoroethylene coated glass fiber, and specifications for silicone varnished glass fiber are being prepared.

Active work is in progress on methods for testing vinyl glass fiber sleeving, thermosetting tapes, and high temperature pressure sensitive tapes.

An extensive round-robin test program is in progress on thermal stability of varnished fabrics, covering a variety of materials.

Subcommittee VIII on Insulating Papers (H. A. Anderson, chairman).—Specifications for Electrical Insulating Paper, Sulfate (Kraft) Tissue for Capacitor Dielectric have been prepared and recommended for publication as information as noted earlier in this report.

Section A on Chemical Tests has carried on extensive collaborative tests on detection of chlorides in insulating papers. Present data show wider deviations than are acceptable and further work is planned.

Section B on Physical Tests has compared a grip type friction test with an inclined block sliding plane test. The latter was found preferable and will be included in the Methods of Sampling and Testing Untreated Paper Used for Electrical Insulation, D 202 - 55 T as a future revision. Other round-robin tests conducted by Section B are on dissipation factor of paper and on felt hair cuts in paper.

Subcommittee IX on Mica Products (Kenneth Wechsler, chairman) sponsored a revision of the Tentative Methods of Testing Pasted Mica (D 352 - 55 T) as noted earlier in this report.

Tentative Specifications for Natural Muscovite Mica Based on Visual Quality (D 351 - 53 T) and Tentative Specifications for Natural Block Mica and Mica Films Suitable for Use in Fixed Mica-Dielectric Capacitors (D 748 - 54 T) are currently being revised. Standard samples of commingled block mica, illustrating defects described in Specifications D 351 - 53 T were prepared and approved.

Tentative methods for testing reinforced pasted mica have been drafted and a suggested method for testing thermal stability of pasted mica is being developed. Plans have been made to develop tentative methods for testing raw mica paper.

Subcommittee X on Magnet Wire Insulation (K. N. Mathes, chairman) held its first meeting at the Benjamin Franklin Hotel in Philadelphia, Pa., on February 15, 1956. Organization into three sections was proposed, and organization was started as follows:

A. Film Coated Magnet Wire (including Oxide),
B. Fibrous Covered Magnet Wire (including Paper),
C. Thermal Capability.

Items proposed for study in each of the sections were solvent capability, soldering and stripping characteristics, mechanical and electrical properties, and continuity and concentricity of insulation.

The activities will be coordinated with those of other organizations concerned with magnet wire.

H. A. Anderson and K. N. Mathes, of Committee D-9, have been appointed to represent ASTM on ASA Project C9, Sectional Committee on Magnet Wire.

Subcommittee XI on Significance of Tests (C. L. Craig, chairman) has revised the Proposed Recommendations for Writing Statements on the Usefulness of Tests of Electrical Insulating Materials (Appendix II of the Compilation of Standards on Electrical Insulating Materials). The revisions have been approved by letter ballot of the subcommittee and by the committee officers and appears in revised form in the 1956 edition of the compilation.

A Symposium on Minimum Property Values of Electrical Insulating Materials[7] sponsored by the subcommittee was presented at the February 14, 1956, meeting of the committee in Philadelphia. C. L. Craig arranged the program and served as chairman.

The subcommittee is working on definitions of electrical insulation terms.

Subcommittee XII on Electrical Tests (N. W. Edgerton, chairman).—Revisions of two tentative methods sponsored by the Subcommittee have been approved by the Administrative Committee on Standards, as noted earlier in this report: Tentative Methods D 495 - 48 T and D 1389 - 56 T.

The subcommittee has also recommended an editorial change in methods D 149 - 55 T, as noted previously in this report.

A Symposium on Corona Measurement and Corona Resistance,[8] sponsored by the subcommittee, was held on

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[7] Issued as separate publication, *ASTM STP No. 188.*

[8] Issued as separate publication, *ASTM STP No. 198.*

November 17, 1955, during the meeting in Cincinnati, Ohio. C. W. Ross arranged the program and served as chairman.

Subcommittee XIV on Conditioning (G. H. Mains, chairman).—The subcommittee is recommending for approval, by joint letter ballot of Committees D-9 and D-20, a revision of the Standard Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (D 618 – 54), as noted above.

Also by joint letter ballot of Committees D-9 and D-20, the subcommittee is recommending that Tentative Specification for Enclosures and Servicing Units for Tests Above and Below Room Temperature (D 1197 – 54 T) be adopted as standard as indicated above.

Special Subcommittee on Technical Papers (L. B. Schofield, chairman) reports the following symposium plans:

In the Spring of 1957, a symposium on Electrical Insulating Oils is scheduled for presentation under the sponsorship of Subcommittee IV.

Projected future symposia are:

"Insulating Oils and Paper for High Voltage Cables"—Subcommittee IV (Spring, 1958); and "Significance of Electrical Test Methods"—Subcommittee XI (Fall of 1957 or 1958).

This report has been submitted to letter ballot of the committee, which consists of 199 members; 149 members returned their ballots, of whom 135 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

A. H. SCOTT,
Chairman.

J. E. GIBBONS,
Recording Secretary.

APPENDIX

DISSIPATION FACTOR AND DIELECTRIC CONSTANT *VERSUS* FREQUENCY FOR FOUR POPULAR INSULATING MATERIALS

By Harry R. Meahl[1]

In 1951, round-robin tests were undertaken by Section C of Subcommittee XII to obtain data on the dissipation factor and dielectric constant of polyvinyl butyral, mica-filled phenolic, cross-linked polystyrene, and Teflon over the frequency range of 100 to 10,000 Mc per sec. It was expected that the effect of frequency on attainable accuracy would be shown.

The results are unexpected. Instead of consistent data at low frequencies and increasing scattering with increasing frequency, the most consistent data were obtained at a particular high frequency, 3000 Mc, and the greatest scattering also occurred at a particular frequency, 100 Mc (the lowest frequency of direct interest since the tests concerned methods used at frequencies above 100 Mc).

Since the results are unexpected, the means by which they were obtained should be examined with a view to directing future investigations in this field.

The usual method of circulating one set of samples to all participants could not be used for two reasons: first, a sample of the proper size and shape to test at 10,000 Mc was not satisfactory for use at other frequencies, and, second, the different participants had measuring equipments for which different sizes and shapes of samples were needed even at the same frequency. For example, equipments operating at 3000 Mc used three shapes of samples: disks, rectangular parallelopipeds and hollow cylinders.

Therefore, each participant was provided with enough material to make a set of samples for his measuring equipments. Four materials were used: polyvinyl butyral (a clear greenish-yellow resin), mica-filled phenolic plastic (Bakelite BM262), cross-linked polystyrene (Textolite 1422—a transparent material), and polytetrafluoroethylene (Teflon—an opaque bluish-white material).

Test Data

The average values obtained are listed in Table I.

The detailed results at 100 Mc and at 3000 Mc are presented in Table II.

Discussion of Data:

The data in Table I for frequencies of 100 Mc and higher are not as consistent as one would like to see them, but probably are typical of measurements in this field. The data for frequencies below 100 Mc are included for comparison. They show satisfactory continuity in measurements of dielectric constant, ϵ, on all four materials and unsatisfactory continuity in measurements of dissipation factor ($\tan \delta$) of polystyrene and Teflon; that is, of very low values.

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[1] General Engineering Laboratory, General Electric Co., Schenectady, N. Y

The data of Table II show that the measurement equipment and techniques used at 3000 Mc have the same order of precision as those used at 100 Mc; that is, the extremely high frequency is not a disadvantage from the measurement point of view.

A review of results of round-robin tests performed in 1940 reveals that progress has been made because the same order of agreement was reached in 1951 making measurements over the frequency range of 100 to 10,000 Mc as had been reached in 1940 over the frequency range of 1 kc to 100 Mc.

TABLE I.—AVERAGE VALUES.[a]

Number of Observers	Frequency, Mc	Polyvinyl butyral		Mica-Filled Phenolic		Cross-linked Polystyrene		Teflon	
		ϵ	Tan δ	ϵ	Tan δ	ϵ	Tan δ	ϵ	Tan δ
1.........	0.0001	3.30	0.0056						
1.........	0.001	3.27	0.0066	5.27	0.0271	2.53	0.00022	2.04	0.00006
1.........	0.01	3.22	0.0129						
1.........	0.1	3.14	0.0256						
1.........	1	3.00	0.0284	4.74	0.0099	2.35	0.00027	2.00	0.00013
1.........	3	2.95	0.0314						
1.........	10	2.84	0.0321	4.74	0.0092	2.35	0.00045	1.96	0.00055
1.........	30	2.78	0.0248	4.63	0.0102	2.56	0.00055	2.03	0.00020
1.........	65	2.77	0.0343						
4.........	100	2.74	0.0248	4.63	0.0110	2.55	0.00057	2.03	0.00028
1.........	150	2.73	0.0102						
1.........	200	2.70	0.020	4.75	0.0130	2.54	0.00051	2.02	0.00035
2.........	300	2.67	0.0255	4.63	0.0162	2.50	0.00071	2.02	0.00036
1.........	400	2.67	0.019	4.74	0.0135	2.55[b]	0.00070[b]	2.03	0.00058
1.........	500	2.72	0.0176			2.55	0.00061	2.06	0.00033
1.........	1000	2.65	0.0217	4.59	0.0160	2.55	0.00050	2.03	0.00033
5.........	3000	2.60	0.0167	4.49	0.0133	2.58	0.00064	2.03	0.00041
2.........	10000	2.65	0.0141	4.60	0.0128	2.54	0.00132	2.06	0.00044

[a] The above values are averages of all observers except for the value of tan δ for mica-filled phenolic at 100 Mc for which one observer's value was omitted because it was ten times those of the other three.

[b] Actually 387 Mc.

CONCLUSIONS

The following conclusions appear reasonable:

1. The quality of measurements is independent of frequency up to 10,000 Mc.
2. Polyvinyl butyral resin has a marked frequency characteristic in both ϵ and tan δ.
3. Mica-filled phenolic plastic has a marked frequency characteristic in both ϵ and tan δ over the range of 100 cps to 1 Mc, but is nearly independent of frequency between 1 and 10,000 Mc.
4. Cross-linked polystyrene and Teflon are independent of frequency with regard to ϵ and tan δ but seem to have a marked frequency characteristic with regard to tan δ below 10 Mc.
5. Teflon has an ϵ 20 per cent lower than that of cross-linked polystyrene.
6. Teflon has a tan δ approximately one-half that of cross-linked polystyrene.

RECOMMENDATION

In future round-robin tests, an effort should be made to get each participant to evaluate the sources of error in his equipment and technique.

TABLE II.—DETAILED RESULTS.

100 Mc						3000 Mc					
ϵ	Maximum Spread, per cent	Average Deviation from Mean, per cent	Tan δ	Maximum Spread, per cent	Average Deviation from Mean, per cent	ϵ	Maximum Spread, per cent	Average Deviation from Mean, per cent	Tan δ	Maximum Spread, per cent	Average Deviation from Mean, per cent
Polyvinyl butyral											
2.91	12.4	3	0.0253	45	15.7	2.58	2.9	1.2	0.0189	27	6.5
2.75			0.032			2.64			0.0181		
2.57			0.0208			2.58			0.016		
2.72			0.021			2.65			0.0142		
						2.57			0.0161		
Mica-Filled Phenolic											
4.19[a]	13.5	4.8	*0.126*[a]	1000	107	4.46	11	3.1	0.0149	33	10
4.75			0.0125			4.65			0.0129		
4.81			0.0108			4.65			0.0131		
			0.0110			4.51			0.0148		
						4.17			0.0102		
Cross-Linked Polystyrene											
2.60	3.1	0.9	0.00089	100	30	2.48	4.8	1.3	0.00071	30	11.5
2.55			0.00060			2.40			0.00054		
2.52			0.00032			2.51			0.00070		
2.54			0.00047			2.52			0.00057		
						2.49			0.00072		
Teflon											
2.06	2.5	0.9	0.00034	48	16	1.97	4.5	1.1	0.00036	164	42.5
2.01			0.00020			2.06			0.00023		
2.02			0.00034			2.05			*0.00090*[a]		
						2.03			0.00023		
						2.04			0.00034		

[a] Obviously erroneous results are italicized.

Measurements were contributed by:

General Radio Co.
Federal Telephone and Radio Co.
General Electric Co.
  Research Laboratory
  General Engineering Laboratory
Westinghouse Electric Co.
Princeton University
National Bureau of Standards
New York Naval Shipyard
  Material Laboratory

Acknowledgments:

Constructive criticism and encouragement have been received from Messrs. Ivan G. Easton, F. Y. Speight, R. R. Winans, S. I. Reynolds, C. N. Works, and F. A. Muller.

REPORT OF COMMITTEE D-10

ON

SHIPPING CONTAINERS*

Committee D-10 on Shipping Containers held two meetings during the year: in New York, N. Y., on September 23, 1955, and in Atlantic City, N. J., on April 12, 1956.

The following officers and members of the Advisory Committee elected for the ensuing term of two years are as follows:

Chairman, Gordon E. Falkenau.
Vice Chairman, Earl R. Stivers.
Secretary, Robert F. Uncles.
Advisory Committee, A. V. Grundy and C. E. Miller.

The committee consists of 104 members, of whom 36 are classified as producers, 42 as consumers, and 26 as general interest members.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following tentatives, which have stood without revision for two or more years, be continued as tentative:

Tentative Methods of:

Vibration Test for Shipping Containers (**D 999 - 48 T**),
Testing Pallets (**D 1185 - 51 T**),
Test for Water Vapor Permeability of Packages by Cycle Method (**D 1251 - 53 T**), and
Test for Water Vapor Permeability of Shipping Containers by Cycle Method (**D 1276 - 53 T**).

There has been no report of field application to establish the adequacy of Methods D 1185, D 1251, and D 1276. Method D 999 is still under review by a special task group. In addition, Methods D 1251 and D 1276 are now subject to review in the light of inadequacies being revealed by Subcommittee III in the Method of Test for Water Vapor Permeability of Shipping Containers (D 1008 - 51).

REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommends that the Standard Definitions of Terms Relating to Shipping Containers (**D 996 - 50**)[1] be revised by the addition of the following terms and reverted to tentative status:

Blank, Paperboard.—Any die-cut, corner cut, scored, or otherwise partially prepared section of paperboard in the flat to be formed into a box or parts thereof, or further cut and scored.

Boxboard.—A general term designating the grades of paperboard used for fabrication of folding and set-up boxes. Customarily shipped in sheets.

Canvas; Canvas Sling.—A heavy closely woven strong fabric, usually made of cotton, produced in sheets or bands; a sling made of canvas.

Carload.—A railroad term defined in Rule 14, Uniform Freight Classification. Usually a load of one kind of article or commodity or of mixed articles or mixed commodities, the weight of which is equal to, or in excess of the published minimum weights, for the articles or commodities, or mixtures.

Corner Buffer.—Various materials, cut or formed to fit into corners of a container and designed to support or cushion the contents.

Die-Cut.—A method of preparation in which part or container has been cut, slotted and/or scored by custom made dies.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17-22, 1956.

[1] 1955 Book of ASTM Standards, Part 7.

Dividers.—A device, made of various materials, which separates the space within a container into two or more spaces, cells, compartments, or layers. Dividers may be plain, interlocking, scored, horizontal, vertical, or diagonal. The primary purpose of dividers is to separate the articles and/or to furnish cushioning.

Dry Finish.—Term descriptive of the surface of a paper or paperboard which in process of manufacture has not been dampened prior to calendering. Resulting therefrom is a surface which is dull and relatively rough. Material is less dense than "water finish." (See Water Finish.)

Elmendorf Test.—A method of determining the ability of paper, fabric, or other sheet material to resist internal tearing. Testing is by mechanical means and results are evaluated in units of energy indicated by scale and expressed as the force in grams required to tear a specimen of standard size.

Excelsior.—Curled shreds of wood, usually soft woods; may be loose, wadded, or made into pads.

Filler Pads.—Used to fill space; sometimes applied to a soft flexible pad made with various loose filling materials to provide cushioning effects.

Fillers.—Any material or combinations of materials used to fill otherwise unused spaces.

Flange, Fibreboard.—A narrow panel attached to a comparatively wide panel and in use folded at an angle. Usually made of scored fibreboard, but panels may be otherwise joined.

French Wrap.—See Wrapper, Extension.

Incline Impact Tester.—A device for applying controlled impacts to a fully packed shipping container. The device consists of an inclined track having an angle of 10 deg to the horizontal; a rigid backstop or bumper positioned at 90 deg to the incline, and a dolly mounted on wheels which run on the track. The intensity of the impact is varied by the distance of movement from the point of release, up the incline. The device may be operated by hand or by a power driven chain with an automatic release. Known also as the Conbur Incline Impact Tester.

Joined Liner.—Any type of box liner having its ends joined by various means.

Kraft (*Cylinder*).—Kraft paper (manufactured on a cylinder-type machine) having a characteristic grain direction.

Kraft (*Fourdrinier*).—Kraft paper (manufactured on a Fourdrinier-type machine) having only minor grain characteristics.

Liner (*Facing*).—The inner and outer sheet of fibreboard.

Macerated Paper.—Generally waste paper torn up mechanically for use as a cushioning material.

Molded Pulp.—Wet paper formed to predetermined shape and then dried. Used for packaging of such items as eggs, fruit, and fragile articles.

Neck, Paperboard.—A sheet or tube inserted in base of package and extending above base into lid when package is closed. Held in position by adhesive or friction.

Nest.—A group of articles, cans, baskets, or boxes, of various sizes, which fit one within the other.

Pail.—A vessel commonly circular in section with or without a bail handle, and often fitted with a cover.

Panel.—Any area (flat) between scores and/or edges of a box, carton, or interior part.

Paperboard.—A broad term designating any paper stock, ranging from 0.009 to 0.030 in. in caliper, used for fabricating containers or components thereof. Used also for various purposes not related to packaging.

Retainer.—A term applied to any device which prevents egress of an article from its intended position.

Score.—Impression or crease in paperboard to facilitate folding. Set-up boxes made of nonbending paperboard are generally partially cut through at score lines and may be reinforced by gummed paper stays.

Separator.—Any material interposed between articles or packing components.

Shell.—A form of various materials enclosing an item; may be entirely closed or open at one or both ends, square, cylindrical, or conforming to the shape of the item. Synonymous with Sleeve (which see). Also applied to unlined caps for containers.

Sleeve.—A form of various materials, usually open at both ends, made in various shapes, which are slipped over an item.

Spacer.—A device made of any material which serves to maintain a predetermined distance between any two points in a shipping container, or between any interior part and articles contained therein.

Spring Pad.—A term loosely used to describe any piece of multipaneled (3 or more) corrugated fibreboard scored and reverse folded alternately and which exerts a spring action when compressed.

Stay, Paper or Metal.—Material used to join parts of a box together at a corner or to reinforce a cut-score on solid paperboard. Most commonly used for set-up boxes.

Step, Paperboard.—Term applied to trays, shells, or platforms positioned inside a container to raise one portion of contents higher than others. May be attached to a lid for purpose of holding certain articles in position when lid is in closed position.

Stitching.—The process of effecting manufacture or closure of containers or parts, and assembly of containers by means of metal fasteners, thread or cordage.

Stop, Paperboard.—Pieces of paperboard attached inside of ends of lid to hold it in proper position when depth of base is less than lid. Commonly used in cosmetic boxes of solid board and millinery and flower boxes made of corrugated board.

Tension Test.—A test to determine the force required to break a strip of paper, fibreboard, wood, metal, wire, or other materials when subjected to pulling force.

Tray.—A shallow open form, with a low rim, used as a space filler or as a support, in interior packing. May have two or more flanges, and be used horizontally, vertically or inverted.

"V" Board.—A form of weather-resistant container board made in solid and corrugated form. Components are heavily sized, dense and highly water resistant. Refer to current government specification for construction details and performance requirements. Used primarily for export or other similar severe service involving possible exposure to weather.

"W" Board.—A form of container board having characteristics similar to "V" board, but meeting lower test requirements. Used previously for interior packing in export shipments. Refer to current government specification.

Water Finish.—Term descriptive of the surface of paper or paperboard which, in process of manufacture, has been dampened on one or both sides prior to calendering.

Wet Strength Paper.—Papers treated chemically to provide a retention of physical characteristics, such as tensile strength, tearing resistance, etc., when the paper is in a damp or wet condition as compared to the same characteristics of the paper when dry.

Wood Wool.—Finely shredded excelsior free from knots, splinters and coarse pieces; plain, wadded or in pads.

Wrapper, Extension.—The extension of the liner beyond the ends of the corrugations on a sheet of single faced corrugated board.

Wrapper, Paperboard.—Generally single faced corrugated used to wrap or sleeve articles prior to placing in a shipping container.

Unit Load.—A plurality of containers or articles secured as a group by a binding or bonding medium; the containers or articles may be on pallets or skids.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Definition of Terms (G. S. Mustin, chairman) prepared the 56 definitions set forth above for inclusion in Definitions D 996 – 50. Additional groups of terms are under consideration.

Subcommittee II on Methods of Testing (J. G. Turk, chairman),

Subcommittee IV on Performance Standards (C. R. Gustafson, chairman), and

Subcommittee V on Correlation of Tests and Test Results (R. C. McKee, chairman).

These three subcommittees continued to function as a joint committee consisting of four task groups, each investigating separate test methods, whose reproducibility has been in question. Although further collaborative work has yet to be accomplished before these task groups can be discharged, the revolving drum test, drop test, and vibration test groups are now arriving at a basis for the revision of their respective test methods (D 782, D 775, and D 999). The stacking test group is writing a method of test for stacking of shipping containers, based on its collaborative test program.

Subcommittee III on Water Vapor Re-

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[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

sistance (H. A. Bergstrom, chairman) has completed a second cooperative test program in connection with its study of the Method of Test for Water Vapor Permeability of Shipping Containers (D 1008 - 51). It is believed the importance of several factors not specified in the present standard have been established and a third collaborative test program, incorporating these specific instructions, will be conducted as a final check before drafting a revision of the standard. It is anticipated that related standards will be reviewed following the conclusions of this investigation.

Subcommittee VI on Interior Packing (S. L. Swenson, chairman) established that the characteristics of cushioning of primary interest were (1) resilience, (2) dusting and breakdown under repeated impact, (3) method of evaluation for use as dunnage, and (4) reaction with other materials and finishes. Task groups were appointed to explore these areas. The task group on Dynamic Testing reported considerable activity in its program aimed at developing a satisfactory test method for dynamic load deflection properties of cushioning materials. Work continues on the compilation of a bibliography of cushioning studies.

This report has been submitted to letter ballot of the committee, which consists of 104 members: 56 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

G. E. FALKENAU,
Chairman.

R. F. Uncles,
Secretary.

REPORT OF COMMITTEE D-11
ON
RUBBER AND RUBBER-LIKE MATERIALS*

Committee D-11 on Rubber and Rubber-Like Materials and eighteen of its subcommittees held meetings in Buffalo, N. Y., on February 29 to March 2, 1956, during ASTM Committee Week. Some of the subcommittees also have held separate meetings during the year, and the SAE-ASTM Technical Committee on Automotive Rubber, with its various sections, has met in Detroit, Mich., on a quarterly basis.

The committee has continued its activity in the work of ISO Technical Committee 45 on Rubber of the International Organization for Standardization. This international group met in Düsseldorf, Germany, September 5 to 10, 1955, and Committee D-11 was represented by R. D. Stiehler (chairman of the American delegation), National Bureau of Standards; R. S. Havenhill, St. Joseph Lead Co.; and R. F. Shaw, U. S. Dept. of the Army, Rock Island Arsenal. Agreement has been reached on a method for determining the indentation hardness of rubber which is recommended later in this report for publication as tentative, and work is in progress on standardization of numerous other methods of testing rubber and latices.

In connection with the coordination of its activities with those of other ASTM technical committees, Committee D-11 has made several changes in liaison representatives during the year: S. S. Kurtz, Jr., Sun Oil Co., was appointed to represent the committee on Technical Committee D-16 on Industrial Aromatic Hydrocarbons, replacing J. F. Anderson who resigned. Because of increasing use of elastomeric plastic materials in insulated wire and cable, E. G. Driscoll, Harbishaw Wire and Cable Div., was appointed as an additional representative on Committee D-20 on Plastics. W. P. Tyler has been appointed to a new Subcommittee on Microchemical Apparatus of Committee E-1. H. G. Bimmerman, E. I. du Pont de Nemours & Co., Inc., G. H. Barnes, Goodyear Tire and Rubber Co., and C. S. Yoran, Brown Rubber Co., Inc., were appointed to represent Committee D-11 on the newly-formed ASTM Coordinating Committee on Cellular Materials.

ASTM is endeavoring to improve coordination of its activities with the standardization work in progress in various departments of the Federal Government and L. V. Cooper, Firestone Tire and Rubber Co. has been appointed to serve as liaison for the committee in this connection.

The officers elected for the ensuing term of two years are as follows:

Chairman, Simon Collier.
Vice Chairman, H. G. Bimmerman.
Secretary, J. J. Allen

Recommendations Accepted by the Administrative Committee on Standards

Subsequent to the 1955 Annual Meeting, Committee D-11 presented to the Society through the Administrative Committee on Standards the following recommendations.

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* **Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.**

Revisions of Tentative Methods for:

Chemical Analysis of Rubber Products (D 297 - 54 T).

Revision of Tentative Method of Test for:

Indentation of Rubber by Means of a Durometer (D 676 - 49 T),
Resistance to Accelerated Light Aging of Rubber Compounds (D 750 - 55 T),
Viscosity of Rubber and Rubber-Like Materials by the Shearing Disk Viscometer (D 927 - 53 T),
Curing Characteristics of Vulcanizable Mixtures During Heating by the Shearing Disk Viscometer (D 1077 - 49 T), and
Accelerated Ozone Cracking of Vulcanized Rubber (D 1149 - 51 T).

Revision of Tentative Specifications for:

Elastomeric Compounds for Automotive Applications (D 735 - 54 T), and
Sheet Rubber Packing (D 1330 - 54 T).

The revision of Method D 1149 was accepted by the Standards Committee on January 23, 1956, the remaining recommendations were accepted on December 19, 1955. The revised methods and specifications appear in the 1955 Book of ASTM Standards, Part 6, bearing the designations indicated above, and in the May, 1956 compilation of ASTM Standards on Rubber Products.

On May 2, 1956, the following recommendations submitted by Committee D-11 were accepted by the Administrative Committee on Standards:

New Tentative Method of Test for:

Stress Relaxation of Vulcanized Rubber in Compression (D 1390 - 56 T).

Revision of Tentative Specifications for:

Rubber Insulating Sleeves (D 1051 - 49 T), and
Construction of Rubber Insulated Wire and Cable (D 1350 - 54 T).

Revision of Tentative Recommended Practice for:

Conditioning of Elastomeric Materials for Low-Temperature Testing (D 832 - 46 T).

This new method of test and the revised specifications and recommended practice appear in the 1956 Supplement to Book of ASTM Standards, Part 6.

The revision of the Tentative Methods for Chemical Analysis of Rubber Products (D 297) adds needed procedures for the determination of iron in crude rubber. The change in the Tentative Method of Test for Indentation by Means of a Durometer (D 676) adds a paragraph on the number of readings and the manner of taking them. This improvement defines the standard measurement as the median of a minimum of five readings. In the Tentative Method of Test for Resistance to Accelerated Light Aging of Rubber Compounds (D 750), the black-panel temperature of 145 ± 5 F previously adopted as standard was found to be unsuitable for certain products such as automobile floor mats. Addition of a note permitting use of other temperatures when required, provides the necessary flexibility in the method. The revisions of the Tentative Methods of Test for Viscosity of Rubber and Rubber-Like Materials by the Shearing Disk Viscometer (D 927) and for Curing Characteristics of Vulcanizable Mixtures during Heating (D 1077) incorporate changes in the methods and the Mooney Viscometer, which resulted from extensive investigations of the National Bureau of Standards carried out in connection with the Government Synthetic Rubber Program. The changes permit use of radial grooved dies, provide for improved temperature measurement, and give precautions when testing high molecular weight materials.

The revision of the Tentative Specifications for Elastomeric Compounds for Automotive Applications (D 735) simplifies the title and changes the scope accordingly. The sections dealing with the testing of finished products were rewritten and rearranged for better clarity. Asterisks were again added to indicate the most frequently used grades in Tables I, III, and IV, and four new silicone grades were added in Table V. In the Tentative Specifications for Sheet Rubber Packing (D 1330), the revision provides for an additional grade of commercial packing which is widely used and slightly raises the quality of the present grade I materials.

The revision of the Tentative Method of Test for Accelerated Ozone Cracking of Vulcanized Rubber (D 1149) changes the scope to incorporate a statement concerning limitations in reproducibility and use for purchase specifications previously given in a note. Work is in progress to improve the method though in its present form, it is considered the best available and is widely used.

The new Tentative Method of Test for Stress Relaxation of Vulcanized Rubber in Compression (D 1390 - 56 T) describes a procedure for measuring a very significant property in evaluating gasket materials. The method has been used successfully for several years by the Materials Laboratory of the New York Naval Shipyard where it was developed and it has been found to have satisfactory reproducibility in other laboratories.

The revision of the Tentative Specifications for Rubber Insulating Sleeves (D 1051) provides for additional sizes and styles of sleeves in line with present commercial practice. Some of the test procedures are also clarified and aging requirements are slightly modified. The revision of the Tentative Specifications for Construction of Rubber Insulated Wire and Cable (D 1350) makes mandatory the use of a protective tape between metallic shields and insulation and also brings the thickness of various nonmetallic sheaths into agreement.

The Tentative Recommended Practice for Conditioning of Elastomeric Materials for Low-Temperature Testing (D 832) has been completely rewritten to incorporate the results of research during the past ten years in the field of low-temperature behavior of rubber. This revision was much needed and brings the recommended practice in line with present knowledge.

NEW TENTATIVES

The committee recommends that the following methods of test and recommended practices be accepted for publication as tentative as appended hereto.[1]

Tentative Method for Tension Testing of Rubber O-Rings:[2]

This method is needed for reference in specifications for O-rings currently in use. It is considered impractical to incorporate the method in the general Methods for Tension Testing of Vulcanized Rubber (D 412). Also, this new tentative may later be expanded to include other tests for this particular rubber product.

Tentative Method of Test for International Standard Hardness of Vulcanized Natural and Synthetic Rubbers:

This method has been developed by the ISO Technical Committee 45 and uses a dead-weight indentation hardness instrument originally employed by the British Admiralty. The equipment has been modified and now has a scale which agrees throughout its range within 2 points with that of the Shore type A durometer (see Method D 676). The method has been accepted by the ISO member bodies including the United States representatives, and Committee D-11 now requests its acceptance by ASTM.

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[1] These new tentatives, with the exception of the Methods of Testing O-Rings, appear in the 1956 Supplement to Book of ASTM Standards, Part 6.

[2] In view of the negative votes cast in Committee D-11 on the acceptance of this method it was withheld from publication.

Tentative Methods for Chemical Analysis of Synthetic Elastomers (Solid Butadiene-Styrene Copolymers):

These methods include a method of sampling and eight test procedures for chemical characteristics of synthetic elastomers. The methods developed originally in the Government synthetic rubber program, have been reviewed and revised where necessary by Subcommittee XIII and are recommended for publication by ASTM.

Tentative Methods of Testing Synthetic Rubber Latices (Butadiene-Styrene Copolymers):

These methods include procedures for sampling and for determining total solids, residual styrene, pH, surface tension viscosity, coagulum, and bound styrene. The basic procedures, developed in the government synthetic rubber program, have been reviewed and revised by Subcommittee XIII, and are recommended for publication by ASTM.

Tentative Recommended Practice for Nomenclature for Synthetic Elastomers and Latices:

This tentative recommends (*1*) the designation "Styrene Rubbers" and the abbreviations "SR" and "SR Latex" to replace the term GR-S for those GR-S type rubbers which contain styrene; and (*2*) the designation "Butadiene Rubbers" and the abbreviations "SBR" and "SBR Latex" to replace the term GR-S for those GR-S type rubbers which contain butadiene only. This is proposed by Subcommittee XIII because the term "Government Rubber-Styrene," (GR-S) is now obsolete.

Tentative Recommended Practice for Description of Types of Styrene Butadiene Rubbers (SBR):

Included in tabular form are the properties of various synthetic polymers according to numbered types. These numbers are the same as those used by the Office of Rubber Reserve, Reconstruction Finance Corp., in the government program. The table is not intended to serve as a specification but merely to establish types of synthetic rubbers to avoid confusion between producers and users. It is believed that this will be helpful, especially during the present transition period.

Tentative Recommended Practice for Description of Types of Styrene Butadiene Rubber (SBR) and Butadiene Rubber (BR) Latices:

This tentative corresponds to the previous one covering the solid polymers and has been developed for the same reasons.

Tentative Recommended Practice for Interlaboratory Testing of Rubber and Rubber-Like Materials:

This tentative gives the concepts and basic principles necessary for design of interlaboratory test programs from which data can be analyzed statistically. It has been developed by Subcommittee XXVIII and is expected to improve the significance of round-robin testing programs.

REVISIONS OF TENTATIVES

The committee recommends that the following five tentatives be revised as indicated and continued as tentative:

Tentative Methods of Test for Adhesion of Vulcanized Rubber to Metal (D 429 - 55 T):[3]

Change Method A to read as appended hereto.[1]

This revision changes the thickness of rubber in the standard ASTM test specimen from $\frac{1}{2}$ in. to $\frac{1}{8}$ in., serving to reduce the deformation of the rubber during test and presumably to lessen the introduction of cross forces which affect the adhesion results. The thinner specimen is an improvement as indicated by results of several round-robin test programs.

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[3] 1955 Book of ASTM Standards, Part 6.

Tentative Methods of Testing Rubber and Thermoplastic Insulated Wire and Cable (D 470 - 54 T):[3]

Section 7.—Change Paragraph (*c*) to read as follows:

> (*c*) For all physical tests, specimens except those of polyethylene shall be marked with gage marks 2 in. apart and placed in the jaws of the testing machine with a maximum distance between jaws of 4 in. For polyethylene specimens, the gage marks shall be 1 in. apart and the distance between jaws 2 in.

Section 36.—Change the second sentence of Paragraph (*a*) to read: "Dry the specimen for 48 hr in a vacuum of 5 mm of mercury or less over calcium chloride at 70 ± 2 C."

Replace the last sentence of Paragraph (*b*) with the following: "Dry the specimen for 48 hr in a vacuum of 5 mm of mercury or less over calcium chloride at 70 ± 2 C. Find the weight, C, of the specimen to the nearest milligram."

These changes provide for the large elongation of polyethylene which otherwise might exceed the test machine travel in the tension test and make uniform the drying procedures in the water absorption test.

Tentative Specifications and Methods of Test for Latex Foam Rubbers (D 1055 - 54 T):[3]

Section 12.—Change the heading, "Low-Temperature Test, Suffix F_1" to read, "Low-Temperature Test, Suffixes F_1 and F_2."

Delete the paragraph headed "Compression-Deflection."

Section 13 (*c*).—Change the first paragraph to read as follows:

> (*c*) When the finished product does not lend itself to testing or to the taking of test specimens because of complicated shape or other reasons, the manufacturer and the purchaser shall agree on the preparation of a suitable test specimen.

Section 14 (*c*).—Change to read as follows:

> (*c*) The steel scale or tape used to measure length or width shall be graduated to $\frac{1}{32}$ in. The dial gage for measuring thickness shall be graduated to 0.001 in. The calipers used for measuring thickness shall be graduated to 0.005 in.

Section 17.—Change the last sentence to read as follows: "The thickness of the test specimen may vary, but shall be not less than $\frac{3}{4}$ in. for slab or uncored stock. The thickness shall be measured and stated in the report."

Section 17.—Change the heading above this section to read: "Compression Set Under Constant Deflection. (Calculations are Based on Amount of Deflection.)"

Section 22.—At the end of this section, add the following sentence: "The full thickness of the product shall be used."

New Section.—Under "Flexing Test" add a new Section 25, as follows, renumbering the subsequent sections accordingly:

> 25. *Test Specimen.*—The test specimen shall consist of the entire product sample or a suitable portion of it as agreed upon by the manufacturer and the purchaser. The full thickness of the product shall be used.

Section 26.—Change the heading above this section to read: "Low-Temperature Test (Suffix F_1 − 40 F) (Suffix F_2 − 65 F)."

Section 27.—Change to read as follows:

> 27. Cylinders 1.129 in. in diameter shall be used for this test. The minimum thickness shall be $\frac{3}{4}$ in. The thickness shall be measured and recorded. Specimens shall be dried in a desiccator for not less than 16 hr before testing.

Section 28.—In the second sentence replace the words, "at a temperature of -40 F," with, "at required temperature."

Section 29.—Change the last line to read: "E = deflection at temperature of test."

Tentative Specifications and Methods of Test for Sponge and Expanded Cellular Rubber Products (D 1056 - 54 T):[3]

Section 13.—Change the heading, "Low-Temperature Test, Suffix F_1," to read, "Low-Temperature Test, Suffixes F_1 and F_2."

Delete the paragraph headed "Compression-Deflection."

Section 15.—Change the last sentence to read: "The thickness shall be measured as described in Section 17 (*b*)."

Add the following sentence at the end of the section:

> As stated under the test methods, the minimum thickness of test specimens is ¼ in. Plied-up samples may be used as indicated in the test methods for compression set and compression deflection (See Note 1 under Section 21).

Section 17 (c).—Change to read as follows:

> (*c*) The steel scale or tape used to measure length or width shall be graduated to $\frac{1}{32}$ in. The dial gage for measuring thickness shall be graduated to 0.001 in. The calipers used for measuring thickness shall be graduated to 0.005 in.

Sections 21 and 24.—Add the following two sentences at the end of both sections: "The minimum thickness shall be ¼ in. Thin samples may be plied-up to obtain this thickness, or a standard test slab may be used if agreed upon by the manufacturer and the purchaser (Note)."

Add the following Note 1 after Section 21, and renumber the present note as Note 2.

> NOTE 1.—Using the same compound thin sections (under ¼ in.) do not blow in the same manner as those over ¼ in. The thinner sections are usually higher in compression-deflection and density.

Section 22.—At the end of the Section add the following reference: "(See Note 1 under Section 21)."

Section 24.—Change the heading above this Section to read: "Compression Set Under Constant Deflection (Calculations are Based on Amount of Deflection."

Section 29.—Change the heading above this Section to read: "Low-Temperature Test (Suffix $F_1 - 40$ F) (Suffix $F_2 - 65$ F)."

Section 30.—Change to read as follows:

> 30. Standard test specimens shall be used for this test. The thickness shall be measured and stated in the report. The minimum thickness shall be ¼ in. Plied-up samples are not satisfactory. The specimen shall be dried in a desiccator for 16 hr before testing.

Section 31.—In the second sentence replace the words, "at a temperature of -40 F," with, "at required temperature."

Section 32.—Change the last line to read: "E = deflection at temperature of test."

Table I.—After the column headed "Suffix F_1," add a new column headed "Suffix F_2," which permits a maximum change of 25 per cent for all grades R-10 to R-15 after being subjected to a low-temperature test at -65 F.

The above revisions clarify these two tentatives and make them more specific. Low-temperature tests at -65 F are also added.

Tentative Method of Test for Compressibility and Recovery of Gasket Materials (D 1147 - 53 T):[3]

Section 3.—Add the following to the end of this Section: "If applied to specimens having thicknesses greater than ¼ in., the results shall be regarded merely as indicative. The specimens shall contain no joint or separation within the minimum test area."

Table I.—Add a new first column headed "Procedure" in which the eight

lines in the table are labeled, respectively, A to H. Also, change the heading of the last column to "Total Load (sum of major load and pre-load), lb."

Section 6.—In the second sentence, change the word "full" to "total." In the fourth sentence, change the word "major" to "total."

ADOPTION OF TENTATIVE AS STANDARD WITH REVISION

The committee recommends that the Tentative Method of Test for Plasticity and Recovery of Rubber and Rubber-Like Materials by the Parallel Plate Plastometer (**D 926 – 47 T**)[3] be approved for reference to letter ballot of the Society for adoption as standard, with the editorial revision consisting of conversion to metric units of values given in English units. Sections 2(*a*), 2(*e*), 3(*a*), 3(*b*), and dimensions in Fig. 2. The method has long been used without comment or criticism.

ADOPTION OF TENTATIVE REVISION AS STANDARD

The committee recommends that the tentative revision issued in January, 1948, of the Standard Methods of Test for Indentation of Rubber by Means of the Pusey and Jones Plastometer (**D 531 – 49**)[3] be approved for reference to letter ballot of the Society for adoption as standard. The tentative revision, requiring the reporting of the time interval for readings, should have been incorporated in the method in the 1949 revision.

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption the replacement of the Standard Specifications for Cotton Rubber-Lined Fire Hose for Public and Private Fire Department Use (**D 296 – 49**)[3] by the Revised Specifications for Woven Jacketed Rubber-Lined Fire Hose, appended hereto,[4] and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revision may be referred to letter ballot of the Society.

Adoption is very desirable because wide-spread use of dacron and nylon in fire hose in this country and Canada has rendered obsolete the existing standard for cotton-jacketed hose. The revision will bring the ASTM standard in line with that of the Underwriters' Laboratories. The revised specifications have received thorough study in the Subcommittee on Hose and are considered to represent the best modern practice. The change in title is incident to the replacement of cotton by synthetic fibers in the construction of the hose.

EDITORIAL CHANGE

The committee recommends the following editorial change in the Tentative Methods of Test for Change in Properties of Elastomeric Vulcanizates Resulting from Immersion in Liquids (**D 471 – 55 T**):[3]

Table II.—Add the following Footnote 6:

[6] Reference fuels Nos. 1 and 2 (formerly included in Method D 471 – 51 T) which are still specified in the Specifications for Non-Metallic Gasket Materials for General Automotive and Aeronautical Purposes (ASTM Designation: D 1170) are available from Phillips Petroleum Co., Bartlesville, Okla.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that all tentatives under its jurisdiction, other than those mentioned in this report, be continued as tentative. They have been reviewed by the various subcommittees and many are under consideration for revision. The results of this work will be presented to the Society as it is completed.

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[4] The revised specifications appear in the 1956 Supplement to Book of ASTM Standards, Part 6.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

ACTIVITIES OF SUBCOMMITTEES

R. A. Bonnell, The Goodyear Tire and Rubber Co., has been appointed chairman of Subcommittee I on Mechanical Rubber Hose replacing C. P. Mullen who resigned. R. H. Titley resigned as chairman of Subcommittee IX on Insulating Tape because of retirement and has been succeeded by C. W. Pickells, Consolidated Edison Co. of New York, Inc. In Subcommittee XVIII on Flexing Tests, B. S. Garvey, Jr., resigned as chairman because of his activities as chairman of Subcommittee XIII, and Howard Tangenberg, The B. F. Goodrich Co., has been appointed to replace him. W. J. Dermody, Stokes Molded Products Co., is now chairman of Subcommittee XXIII on Hard Rubber, replacing C. P. Morgan who resigned.

Of the many interesting and important projects in progress in the subcommittees of Committee D-11, only a few can be mentioned here.

Subcommittee I on Hose (R. A. Bonnell, chairman), having completed the revision of the fire hose specification presented in this report, is now well advanced on a revision of the Tentative Methods of Testing Rubber Hose (D 380) which will probably be presented to the Society next year.

Subcommittee V on Insulated Wire and Cable (J. T. Blake, chairman) expects soon to complete revisions of the specifications for neoprene sheaths for wire and cable. The specification for heavy-duty material (D 752) will be restricted to black neoprene jackets, and the physical test requirements of the specification for light-duty material (D 753) will be changed so that it may be used for general-purpose colored neoprene jackets. The subcommittee is also studying proposals to include detailed specifications on knitted coverings, wrapped coverings, and nylon-, rayon- and glass-braid covering in the Wire and Cable Construction Specification (D 1350).

Subcommittee VIII on Nomenclature (H. L. Fisher, chairman) has completed final revision of the Glossary of Terms Relating to Rubber and Rubber-Like Materials. This valuable glossary is being published by the Society as *Special Technical Publication No. 184.*

Subcommittee IX on Insulating Tape (C. D. Pickells, chairman) expects soon to complete revisions in the Specifications for Friction Tape (D 69), for Rubber Insulating Tape (D 119), and for Ozone-Resistant Tape (D 1373). These changes will improve the procedures for sampling and rejection and will make some changes in test methods.

Subcommittee X on Physical Testing (L. V. Cooper, chairman) is considering a proposed tentative method for tension testing of rubber by means of the strain tester developed by the National Bureau of Standards. If approved this will be presented to the Society later through the Administrative Committee on Standards.

Subcommittee XV on Life Tests (G. C. Maassen, chairman) is revising the Standard Method of Test for Resistance to Light Checking and Cracking (D 518 – 44) by the addition of a new method C using a different and improved type of specimen. If completed prior to the next Annual Meeting, this revision will be presented to the Society through the Administrative Committee on Standards as a tentative revision of the standard.

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[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee XXIII on Hard Rubber (W. J. Dermody, chairman) has decided to eliminate its section on asphalt battery containers and to combine those activities with the work on hard rubber. This subcommittee is starting an extensive program on revision of the hard rubber test methods D 530. Among other changes, the addition of a hardness test using the Type D Shore durometer and the substitution of a heat distortion test for the present softening point test are being considered.

Subcommittee XXIX on Compounding Ingredients (A. E. Juve, chairman) is developing a set of recipes for testing carbon blacks in rubber. Two series of round-robin tests have been completed and further work is in progress. The subcommittee is also working on revision of the Tentative Methods of Sample Preparation for Physical Testing (D 15) to incorporate modifications which recent developments have shown to be desirable. The subcommittee has announced that an additional standard reference material—an SRF carbon black—has been added to the list of those provided by the National Bureau of Standards.

This report has been submitted to letter ballot of the committee, which consists of 243 members; 102 members returned their ballots, of whom 88 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

S. COLLIER,
Chairman.

ARTHUR W. CARPENTER,
Secretary.

REPORT OF COMMITTEE D-12

ON

SOAPS AND OTHER DETERGENTS*

Committee D-12 on Soaps and Other Detergents held one meeting during the year: on March 19 and 20, 1956, in New York City.

A resignation regretfully accepted was that of Fred Krassner, Vice-Chairman of the committee and chairman for many years of Subcommittee S-1. Announcement of the loss by death of two valued and highly respected members of the committee, Clyde Hayward and E. A. Robinson, was received with much regret.

On the recommendation of the committee, W. H. Joy has been appointed as an ASTM representative to ASA Sectional Committee K63 on Institutional Cleaning and Maintenance Materials.

M. G. Kramer was appointed chairman of Subcommittee T-5, succeeding A. M. Schwartz.

The officers elected for the ensuing term of two years are as follows:

Chairman. Jay C. Harris.
Vice-Chairman, W. H. Koch.
Secretary, E. W. Colt.

The committee consists of 118 members, of whom 53 are classified as producers, 35 as consumers, and 25 as general interest members, with 5 consulting members.

REVISION OF TENTATIVE

The committee recommends that the Tentative Methods for Sampling and Analysis of Sodium Triphosphate (**D 501 – 55 T**)[1] be revised by the addition of a procedure for determining orthophosphate as appended hereto.[2]

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentative methods be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Method of Test for:

pH of Aqueous Solutions of Soaps and Detergents (**D 1172 – 54 T**),[1] and
Surface and Interfacial Tension of Solutions of Surface-Active Agents (**D 1331 – 54 T**).[1]

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption the following revisions of the Standard Definitions of Terms Relating to Soaps and Other Detergents (**D 459 – 55**),[1] and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Change the definitions for soil, detergent, and detergency to read as follows:

Soil.—Matter out of place.

Detergent.—A composition that removes soil.

Detergency.—The removal of soil using a detergent.

*** Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.**

[1] **1955 Book of ASTM Standards Part 7.**

[2] **The revised tentative appears in the 1956 Supplement to Book of ASTM Standards, Part 7.**

Tentatives Continued Without Revision

The committee recommends that the following methods be continued as tentative without revision:

Tentative Method of:

Test for Buffering Action of Metal Cleaners (**D 1279 - 53 T**),
Total Immersion Corrosion Test for Soak Tank Metal Cleaners (**D 1280 - 53 T**), and
Test for Rinsing Properties of Metal Cleaners (**D 1281 - 53 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

Subcommittee Activities

Subcommittee T-2 on Analysis of Soaps and Synthetic Detergents (J. C. Harris, chairman):

Task Group 1, Problem Advisory Group (C. A. Gerardi, chairman) is considering the current needs for new and revised methods for analysis of soaps and synthetic detergents for the purpose of determining which projects should next be developed.

Task Group 2 on Rapid Spectrophotometric Determination of Copper in Soap (E. W. Blank, chairman).—Cooperative tests made with two samples of milled soaps containing two ranges of copper yielded an unsatisfactory estimate of precision which was attributed to lack of homogeneity in distribution of the copper salt. New analyses are planned on soap to which accurately weighed amounts of copper are added at the time and place of analysis.

Task Group 3 on Bibliographical Abstracts on Analytical Methods for Surfactants[4] (J. C. Harris, chairman).—This bibliography is currently being revised and brought up to date.

Task Group 4 on Determination of Active Ingredient in Unbuilt Surfactants (J. G. Miller, chairman).—Previous considerations indicated use of solvent extraction for alkylaryl sulfonates. Replies to a questionnaire indicated ethanol as the most satisfactory solvent, and Methods of Chemical Analysis of Soaps Containing Synthetic Detergents (D 820) and Method of Analysis of Oil-Soluble Petroleum Sulfonates (D 855) are considered essentially satisfactory for cooperative work.

Task Group 5 on Infrared Determination of Surfactants (P. Sadtler, chairman) is continuing the development of methods for infrared determinations. A variation in technique has been suggested as a means of developing more effective data.

Task Group 6 on Volumetric Method of Total Phosphorus Pentoxide (R. C. Stillman, chairman).—Cooperative tests indicate that this method is superior to gravimetric methods. A standard deviation of 0.20 per cent P_2O_5 was considered realistic for P_2O_5 contents up to 60 per cent.

Task Group 7 on Glycerol in Soap by Periodate Oxidation (C. A. Snell, chairman) has obtained samples of soaps for cooperative test purposes and cooperative testing will begin shortly.

Task Group 8 on Determination of CMC (J. L. Darragh, chairman).—A survey of methods is under way.

Task Group 10 on Sulfate Content in Presence of Phosphates (A. K. Light, chairman).—Preliminary work on this problem has been undertaken.

Task Group 11 on Determination of Active Ingredient by Ultraviolet Absorption (C. W. Parkert, chairman).—Sodium dodecyl benzene sulfonates were prepared from four different dodecyl benzenes by a standard sulfonation procedure. They were analyzed by standard methods and then by ultraviolet analysis at both 224 mμ and 261 mμ. The results were circulated to Task Group 11 for consideration. Similar samples are to be circulated to the members of the task group to obtain comparative data.

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[5] Issued as separate publication, *ASTM STP No. 150-A*.

A background of qualitative ultraviolet data for the three classes of surfactants—anionic, cationic, and nonionic—is to be collected in the near future.

Task Group 13 on Determination of Tripolyphosphate (B. N. Rockwood, chairman).—It is expected that work by this group will be based upon the chromatographic method.

Task Group 14 on Rapid Determination of Moisture by Infrared Lamp (O. L. Sherburne, chairman).—Preliminary work is under way.

Task Group 15 on Determination of Active Ingredient in Soaps Containing Synthetic Detergents by Cationic Titration (L. E. Weeks, chairman).—A survey indicates use of both the *p*-toluidine hydrochloride and the cationic method. Cooperative tests on a standard detergent are under way.

Task Group 16 on Qualitative Method for Identification of Surfactants and Organic Builders (R. J. Steele, chairman).—Preliminary evaluation of possible methods is under way.

Task Group 17 on Critical Review of Methods for Analysis of Inorganic Builders (A. Mankowich, chairman).—The survey indicates the titrimetric method for P_2O_5 as most favorable. Interference by phosphates in determination of sulfates causes erroneous results. Other builder methods are being evaluated.

Task Group 21 on Detergent Alkylate (J. C. Harris, chairman) is considering methods for distillation range, flash point, viscosity, water or moisture, sediment, aniline point, bromine number, refractive index, specific gravity, Saybolt color, and doctor test.

Subcommittee T-3 on Analysis of Dry Cleaning Materials (G. P. Fulton, chairman) is being reorganized.

Subcommittee T-4 on Analysis of Alkaline Detergents (W. H. Koch, chairman).—Collaborative work on the analysis for tripolyphosphate content by the cobalt complex of Weiser has been encouraging but does not warrant adoption without further work.

Task groups have been set up on (1) Assay of Triphosphate by Tris(ethylenediamine) Cobalt (III) Chloride, M. V. Trexler, chairman, and (2) Chromatographic Analysis of Triphosphate, C. F. Callis, chairman.

Subcommittee T-5 on Physical Testing (A. M. Schwartz, chairman):

Task Group 1 on Measurement of Soil Redeposition (M. G. Kramer, chairman).—Cooperative testing is under way. The Annotated Bibliography by L. E. Weeks has been published, and a list of additional references was distributed.

Task Group 2 on Measurement of Wetting Power (C. A. Gerardi, chairman) is investigating the reproducibility of Draves skeins and Shapiro tapes.

Task Group 3 on Measurement of Reflectance of Test Fabrics (R. B. Diaz, chairman).—Cooperative testing is now in progress.

Task Group 4 on Measurement of Rewetting Power (H. J. Ranauto, chairman).—Cooperative testing is being initiated.

Task Group 5 on Brightener Evaluation (O. C. Bacon, chairman).—Cooperative evaluation tests are under way.

Task Group 6 on Methods for Measurement of Detergency (W. J. Hart and E. B. Ashcraft, Co-chairmen).—Preliminary work has been initiated.

The following new task groups have been formed:

Task Group 7 on Radiotracers in Detergency (E. B. Ashcraft, chairman).

Task Group 8 on Methods for Evaluation of Rug Cleaning Detergents (J. W. Rice, chairman).

Task Group 9 on Methods for Determination of Cloud Points of Detergents.

Subcommittee T-6 on Metal Cleaning (H. A. Kafarski, chairman).—A new task group will be formed to evaluate and improve the Methods for Chemical Analysis of Industrial Metal Cleaning Compositions (D 800 – 45).

The task group on Automotive Industries Cleaning has about completed the development of a laboratory test to evaluate detergents for spray power washers. Several new task groups are being organized.

A paper by Harris, Kramer, and Trexler entitled "A Corrosion Test for Vitreous Enamel Surfaces" will appear in the September, 1956, issue of the ASTM BULLETIN. This method will be investigated further.

Subcommittee G-2 on Nomenclature and Definitions (L. T. Howells, chairman) prepared the revision in D 459 – 55 referred to earlier in the report. Consideration has been given to new definitions for precision and accuracy.

Subcommittee G-5, Program Planning (R. B. Mitchell, chairman) was responsible for the program presented at the March meeting which included the following technical papers or presentations:

"Vitreous Enamel Corrosion Test" by J. C. Harris, M. G. Kramer, and M. V. Trexler.

"Some Aspects of Brightener Testing" by H. W. Zussman, W. Lennon, and W. Tobin.

"Measurement of Water-Soluble Soil Removal in Drycleaning" by G. P. Fulton, A. C. Lloyd, F. Loibl, G. Moore, and D. Reichard.

"Detergency Evaluation" by W. Craig White.

A demonstration of ultrasonic cleaning by R. Alan of the Industrial Scientific Products Div. of Curtiss-Wright Corp.

This report has been submitted to letter ballot of the committee, which consists of 118 members; 75 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. C. HARRIS,
Chairman.

E. W. COLT,
Secretary.

REPORT OF COMMITTEE D-13

ON

TEXTILE MATERIALS*

Committee D-13 on Textile Materials held two meetings during the year: On October 18 to 21, 1955, and on March 13 to 16, 1956, both in New York, N. Y.

At the fall meeting, Walter J. Hamburger, Director, Fabric Research Laboratories, spoke on the subject "A Technology for the Analysis, Design, and Use of Textile Structures as Engineering Materials." Dr. Hamburger also presented this material at the 1955 Annual Meeting of the Society in the form of the Marburg Lecture.

Speaking before Subcommittee A-1, Otto Goedecke, President of the Otto Goedecke Co., and chairman, Committee on Fiber Testing and Correlation of the American Cotton Shippers Association, reported on "The Cotton Shipper's Viewpoint." The American Cotton Shippers Assn. has adopted the ASTM cotton fiber testing methods in its official rules governing commercial transactions in cotton.

At the spring meeting, the Harold DeWitt Smith Memorial Medal for 1956 was presented to Arthur G. Scroggie. Retiring Secretary William H. Whitcomb was presented a silver tray in recognition of over a quarter century of service to Committee D-13. This meeting had a record attendance of 202 members and 51 guests. Thirty-five task groups and 18 subcommittees held meetings.

Stanley Backer was appointed to succeed B. L. Whittier as the "educator" member of the Smith Award Committee. The following appointments of subcommittee officers were made during the year: Jackson Bauer, chairman, and Frank O'Neil, secretary of A-3; M. C. Bullock, chairman of A-9; George F. Quimby, secretary of A-5; B. S. Sprague, chairman, and Donald J. Leitgeb, secretary of B-7; W. J. D. van Dobbenburgh, secretary of B-8; T. D. Lewis, Jr., secretary of B-9.

The officers elected for the ensuing term of two years are as follows:

Chairman, B. L. Whittier.
First Vice-Chairman, S. J. Hayes.
Second Vice-Chairman, K. L. Hertel.
Secretary, H. A. Ehrman.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1955 Annual Meeting, Committee D-13 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Methods of:

Testing Spun and Filament Yarns Made Wholly or in Part of Man-Made Organic Base Fibers (D 1380 - 55 T),

Test for Stiffness of Fabrics (D 1388 - 55 T),

Revision of Tentative Specifications for:

Fire-Retardant Properties of Treated Textile Fabrics (D 626 - 55 T),

Revision and Reversion to Tentative of Standard Methods of:

Quantitative Analysis of Textiles (D 629 - 55 T),

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

Withdrawal of Methods of:

- Testing Continuous Filament Man-Made Organic Base Yarns (D 258 - 52 T),
- Testing and Tolerances for Spun Rayon and Acetate Yarns and Threads (D 507 - 44), and
- Testing and Tolerances for Yarns Spun from Mixed Fibers (D 508 - 53).

The new Method D 1388, and the revisions of Specifications D 626 and Methods D 629 were accepted by the Standards Committee on January 4, 1956; the remaining recommendations were accepted on September 9, 1955. The new and revised tentatives appear in the 1955 Book of ASTM Standards, Part 7.

RECOMMENDATIONS AFFECTING STANDARDS

The committee is presenting for publication 1 proposed method as information, 5 new tentatives, revisions of 5 tentatives, tentative revisions of 1 standard, revision and reversion to tentative of 1 standard, adoption of 5 tentatives as standard, revisions for immediate adoption of 2 standards, and editorial revisions of 1 tentative.

The standards and tentatives affected, together with the revisions recommended, are listed in detail in the Appendix.

All other standards and tentatives not specifically referred to are being actively studied by the respective subcommittees and it is recommended that they be continued in their present status.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[1]

This report has been submitted to letter ballot of the committee, which consists of 372 members; 153 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. D. APPEL,
Chairman.

H. A. EHRMAN,
Recording Secretary.

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[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-13 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Tolerances for:

Spun Rayon and Acetate Yarns (D 1453 - 56 T), and
Yarns Spun from Mixed Fibers (D 1454 - 56 T).

These recommendations were accepted by the Standards Committee on September 5, 1956, and the new tentative tolerances appear in the 1956 Supplement to Book of ASTM Standards, Part 7.

APPENDIX I

RECOMMENDATIONS AFFECTING STANDARDS FOR TEXTILE MATERIALS

In this Appendix are given proposed revisions in certain standards and tentatives covering textile materials. These standards and tentatives appear in their present form in the 1955 Book of ASTM Standards, Part 7.

PROPOSED METHOD TO BE PUBLISHED AS INFORMATION

The committee recommends that the Proposed Method of Test for Strength of Cotton Fibers Using a Finite Gage Length (Flat Bundle Method) be accepted for publication as information only, as appended hereto.[1]

NEW TENTATIVES

The committee recommends that the following methods be accepted for publication as tentative, as appended hereto:[2]

Tentative Methods of:

Test for Non-Lint Content of Cotton (Shirley Analyzer Method),
Test for Twist in Single Spun Yarns (Untwist-Twist Methods),
Test for Twist in Single Spun Yarns (Direct-Counting Method),
Test for Unevenness of Textile Strands,
Test for Tear Resistance of Woven Fabrics by Falling-Pendulum Method.

REVISION OF TENTATIVES

The committee recommends that the following five tentatives be revised as indicated and continued as tentative:

Tentative Methods of Quantitative Analysis of Textiles (D 629 – 55 T):

New Section.—Add the following procedure for determining Acrilan in mixtures with wool as a new Section 15 with a new Note 15, renumbering the present sections and notes accordingly:

15. *Acrilan:* (*a*) *Application.*—This procedure is applicable to mixtures of Acrilan with wool. Wool is removed completely by a boiling sodium hydroxide solution (5 per cent), whereas Acrilan acrylic fiber is substantially unaffected by the procedure specified.

(*b*) *Apparatus:*

(*1*) *Pyrex Beaker*, 250-ml capacity.

(*2*) *Glass Stirring Rod.*

(*3*) *Fritted Glass Filtering Crucible*, coarse porosity.

(*4*) *Weighing Bottle*, suitable for containing fritted glass filtering crucible.

(*5*) *Suction Flask* with suitable adapter.

(*c*) *Reagents:*

(*1*) *Sodium Hydroxide Solution* (*5 per cent*).—Dissolve 50 g of sodium hydroxide (NaOH) in 950 g of water.

(*2*) *Acetic Acid Solution* (*5 per cent*).—Dissolve 50 g of glacial acetic acid ($CH_3 \cdot COOH$) in 950 g of water.

(*d*) *Test Specimen.*—A test specimen weighing approximately 1 g shall be analyzed. Remove nonfibrous materials in accordance with the procedure described in Section 5 and determine the oven-dry weight of the clean fiber residue, weight *C*. Before making the analysis cut the specimen into approximately $\frac{1}{4}$-in. squares to facilitate dissolving the wool.

(*e*) *Procedure.*—Immerse the specimen in 100 ml of boiling NaOH solution (5 per cent) and boil for 10 min. Stir the solution frequently during this period and then filter through a fritted glass filtering crucible using suction to remove excess liquid. Wash the fiber residue twice with 100-ml portions of NaOH solution (5 per cent), three times with 150-ml portions of distilled water, twice with 100-ml portions of acetic acid solution (5 per cent) and three times with 150-ml portions of distilled water. Drain with the aid of suction, place the crucible in a weighing bottle and dry the residue to constant weight in an oven at 105 to 110 C, weight N.

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[1] This method appears in the 1956 Compilation of ASTM Standards on Textile Materials.

[2] The new tentative appears in the 1956 Supplement to Book of ASTM Standards, Part 7.

(*f*) *Calculation.*—Calculate the Acrilan content from Eq (22), Section 15 (Note 15).

NOTE 15.—Interlaboratory analyses have shown that this method may be used to determine the Acrilan content of Acrilan-wool blends with a 95 per cent confidence range of ±0.7 per cent for the mean of two determinations.

Section 15.—Add the following new Eq 22 and renumber the present Eqs 22 and 23. Change references to these equations in the text accordingly.

$$\text{Acrilan, per cent} = \frac{N}{C} \times 100 \quad \ldots\ldots (22)$$

Section 16 (*d*).—Renumber the equations.

Tentative Method of Test for Tensile Strength of Wool Fiber Bundles (D 1294 – 53 T):

Revise as appended hereto.[2]

Tentative Methods of Testing and Tolerances for Rayon Tire Cord (D 885 – 54 T):

Section 19(*a*).—Change rate of force from "16 ± 0.5 g per denier per min" to "18 ± 2 g per denier per min."

Tentative Methods of Testing and Tolerances for Yarns Containing Wool (D 1285 – 53 T):

Section 10(*b*).—Change to read as follows:

(*b*) *Worsted Yarn.*—The yarn number of worsted yarn shall be within the following tolerances:

	Yarn Number Tolerances
Single yarn, coarser than No. 23	±6.5
Single yarn, No. 23 and finer	±5.5
Plied yarn, all numbers	±8.0

Note 2.—Change to read as follows:

NOTE 2.—The tolerances here set forth are recommended for use in the arbitration of disputes. These tolerances apply to material in the form of spun yarn only. They are not applicable to yarn in fabric or garments or in any state in which its form has been changed from the spun yarn.

Tentative Methods of Testing Spun End Filament Yarns Made Wholly or in Part of Man-Made Organic Base Fibers (D 1380 – 55 T): Delete all references to the Typp system of yarn number.

TENTATIVE REVISIONS OF STANDARDS

The committee recommends tentative revisions of the Standard Definitions of Terms Relating to Textile Materials (**D 123 – 55**), as follows:

Add the following definitions:

Fiber, Natural, n.—A class name for various genera of fibers (including filaments) of (*1*) animal, (*2*) mineral, or (*3*) vegetable origin.

NOTE: *Examples.*—(*1*) silk and wool, (*2*) asbestos, (*3*) cotton, flax, jute, ramie.

Fiber, Man-made, n.—A class name for various genera of fibers (including filaments) produced from fiber forming substances which may be (*1*) polymers synthesized by man from simple chemical compounds, (*2*) modified or transformed natural polymers, and (*3*) glasses.

NOTE: *Examples.*—(*1*) polyacrylic, polyamide, polyester, polyethylene, polyurethane, and polyvinyl fibers are truly synthetic, being formed from polymers produced by man; (*2*) alginic fibers and cellulose base fibers, such as acetate and rayon, are examples of fibers made from polymers produced by nature.

REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommends that the Standard Methods of Identification of Fibers in Textiles (**D 276 – 49**) be revised as appended hereto[2] and reverted to tentative status.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following five tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Method of Test For:

Maturity of Cotton Fibers (Random Sample-Sodium Hydroxide Swelling Method) (**D 1443 - 53 T**),
Cross-Sectional Characteristics of Cotton Fibers (**D 1444 - 53 T**),
Micronaire Fineness of Cotton Fibers (**D 1448 - 54 T**),

Tentative Specification and Methods of Test for:

Fineness of Wool Tops (**D 472 - 55 T**), and

Tentative Methods of:

Core Sampling of Raw Wool in Packages for Determination of Percentage of Clean Wool Fiber Present (**D 1060 - 55 T**).

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption, revisions in the following standards consisting of the deletion of all references to the Typp system of yarn number, and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Methods of:

Testing and Tolerances for Cotton Sewing Threads (**D 204 - 42**), and
Test for Yarn Number of Yarn from Fabrics (**D 1059 - 52**).

EDITORIAL CHANGE

The committee recommends the following editorial change in the Tentative Methods of Test for Stiffness of Fabrics (**D 1388 - 55 T**):

Section 1(d).—Change to read as follows:

> (*d*) The two methods may not give the same numerical values but both give excellent rank correlation with a subjective evaluation obtained by feeling the fabric.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following specifications and methods be continued as tentative without revision:

Tentative Specifications and Method of Test for:

Fineness of Wool (**D 419 - 55 T**),

Tentative Method of Test for:

Fiber Length of Wool Tops (**D 519 - 55 T**),
Vegetable Matter and Alkali-Insoluble Impurities in Scoured Wool (**D 1113 - 54 T**),
Average Fiber Diameter of Wool Tops by Porous Plug Tester (**D 1282 - 53 T**),
Wool Content of Raw Wool (Commercial Scale) (**D 1334 - 54 T**), and

Tentative Method of Sampling and Testing:

Staple Length of Wool in the Grease (**D 1234 - 54 T**).

REPORT OF COMMITTEE D-14

ON

ADHESIVES*

Committee D-14 held two meetings during the year: at Case Institute of Technology, Cleveland, Ohio, on October 27 and 28, 1955, and in Louisville, Ky., on April 4, 5, and 6, 1956. The third day of the April meeting was devoted to field trips to Joseph E. Seagram & Sons, Inc., to observe the use of adhesives for labeling and packaging, and to the Mengel Co. furniture and plywood plant.

The committee consists of 133 members, of whom 49 are classified as producers, 59 as consumers and 25 as general interest or consulting members.

The following subcommittee chairmen have been appointed:

Subcommittee II on Analytical Properties, G. F. Lipsey.

Subcommittee IV on Working Properties, N. J. DeLollis.

Subcommittee VI on Nomenclature, G. W. Koehn.

Subcommittee VIII on Electrical Properties, F. Moser.

Subcommittee IX on Editorial Review under the chairmanship of Frank W. Reinhart was established at the October meeting.

A section on Stress Concentration was established under the jurisdiction of Subcommittee I. Three task groups on Slippage and Flow, New Applications of Adhesives, and New Methods of Evaluation of Adhesives were established under the jurisdiction of Subcommittee VII.

The officers elected for two-year terms were as follows:

Chairman, R. F. Blomquist.

Vice-Chairman, J. E. Rutzler, Jr.

Secretary, J. J. Lamb.

Membership Secretary, W. W. Sederlund.

REVISIONS OF TENTATIVES

The committee recommends that the Method for Determining the Effect of Moisture and Temperature on Adhesive Bonds (**D 1151 - 51 T**), be revised as follows and continued as tentative:

Table I.—In Footnote (*b*), delete the words, "Or, alternatively immersion in water at room temperature (70 to 100 F) for a 48-hr period or until equilibrium (constant weight) is reached," and after the words "vacuum at 20" insert "±1."

Section 4(*b*).—Place a period after the words "Table I," and delete "unless otherwise specified by written agreement between the manufacturer and the purchaser of the adhesives."

Section 7.—In Paragraph (4), after the words "Test conditions used" insert the phrase "any deviation from conditions listed in Table I."

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following two tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

Method of Test for:

Storage Life of Adhesives by Consistency and Bond Strength (**D 1337 - 54 T**),[1] and

Working Life of Liquid or Paste Adhesives by Consistency and Bond Strength (**D 1338 - 54 T**).[1]

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following tentatives, which have been published by the Society for two or more years, be continued without revision pending the results of further study:

Tentative Recommended Practice for:

Determining the Effect of Artificial (Carbon-Arc Type) and Natural Light on the Permanence of Adhesives (**D 904 - 46 T**), and

Determining Strength Development of Adhesive Bonds (**D 1144 - 51 T**).

Methods of Test for:

Strength Properties of Adhesives in Shear by Tension Loading (Metal-to-Metal) (**D 1002 - 53 T**),

Effect of Mold Contamination on Permanence of Adhesive Preparations and Adhesive Bonds (**D 1286 - 53 T**), and

Adhesives Relative to Their Use as Electrical Insulation (**D 1304 - 54 T**).

Method of Testing:

Cross-Lap Specimens for Tensile Properties of Adhesives (**D 1344 - 54 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Strength Properties (C. E. Britt, chairman).—Current widespread interest in high-strength adhesives in the aircraft and building industries is reflected in the subcommittee activities.

Investigations are under way on rate of loading to be specified in the Test for Strength Properties of Adhesives in Shear by Tension Loading (Metal-to-Metal) (D 1002 - 53 T), evaluation of peel strength of metal-to-metal adhesive bonds and evaluation of creep properties of metal-to-metal joints in long-time loading. Closely related to this problem is the determination of the suitability of chambers used to conduct strength tests of bonded joints at high and low temperatures. A recommended practice covering this latter phase has been prepared.

The versatility of wood as a construction material is recognized, particularly when employed in bonded composite structures employing metals, glass, or rubber. The problems of evaluating such composite materials are being considered, with attention being given to the use of brittle lacquer coatings and electrical strain gages.

Subcommittee II on Analytical Tests (G. F. Lipsey, chairman).—Proposed methods of test for amylaceous matter in adhesives, nonvolatile content of aqueous adhesives and for nonvolatile content, filler content, and hydrogen ion concentration of phenol-, resorcinol-, and melamine-formaldehyde resin adhesives have been developed. Methods for the nonvolatile content and specific gravity of solvent-type adhesives have also been developed.

Subcommittee III on Tests for Permanency (D. A. George, chairman).—Plans have been made for collecting and reviewing information on various exposure and service tests of adhesive bonded wood joints now in progress throughout the country. Efforts will be made to correlate the results of these long-time tests with short-time laboratory permanence tests already developed or under consideration. A study was made of two alternative immersion procedures for the Method of Test for Effect of Moisture and Temperature on Adhesive Bonds

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[1] 1955 Book of ASTM Standards, Part 7.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

(D 1151 – 51 T), resulting in a revision of part of this procedure. The need for additional test methods on the effects of chemical reagents and of microorganisms on adhesive bonds is under consideration.

Subcommittee IV on Working Qualities (M. Petronio, chairman) is considering a stroboscopic control method for effecting constant rate of shear of the Stormer viscometer when used in accordance with the Tentative Method of Test for Consistency of Adhesives (D 1084 – 55 T), Method C.

A report summarizing the data obtained with the modified Interchemical Corp. Tackmeter is being prepared and will be submitted for publication. Another report will summarize the results of a study on critical glue-line thickness. Information from a questionnaire on slippage and flow will be used to guide activities on this subject.

Subcommittee V on Specifications (S. L. Adams, chairman) has undertaken a program to develop an acceptable statistical sampling technique for liquid adhesives. The initial phase of the program is devoted to developing quantitative data which will permit statistical evaluation of variability.

A tentative specification for room-temperature and intermediate-temperature setting wood adhesives has been submitted to letter ballot of the subcommittee and is being reviewed in light of revised military specifications. A tentative specification and method of test for bonding permanency of adhesives for labeling glass bottles has been developed and is being submitted to letter ballot of the subcommittee. Work is continuing on specifications for adhesives for sealing top flaps of shipping cases and on general utility adhesives.

Subcommittee VI on Nomenclature (F. W. Reinhart, chairman) is defining a number of new terms and some have been out for letter ballot. Recent attention has been focused on terms related to adhesives for paper and paper containers.

Subcommittee VII on Research (J. E. Rutzler, Jr., chairman) has initiated a program of preparing semiannual lists of literature references of importance to workers in the fields of adhesion and adhesives.

Considerable attention has been devoted to the fundamentals of tack in order to define, classify, measure, and interpret this property more specifically. New task groups are being organized on the fundamentals of slippage and flow properties, and to assemble and consider new applications of adhesives and new methods for evaluating adhesive bonds. During the year, research papers were presented on the following subjects: "Progress in the Study of Tack," by F. H. Wetzel; "Influence of Polymer Orientation at Interfaces on Joint Strength," by H. R. Merriman; "Adhesion of Polyethylene to Metals," by N. J. DeLollis; "Forces Involved in Adhesive Bonds," by J. E. Rutzler, Jr.

This report has been submitted to letter ballot by the committee, which consists of 133 voting members; 71 members returned their ballots, of whom 62 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

R. F. Blomquist,
Chairman.

C. K. Merrill Winne,
Secretary.

REPORT OF COMMITTEE D-15

ON

ENGINE ANTIFREEZES*

Committee D-15 on Engine Antifreezes held two meetings during the year: the first in New York, N. Y., on October 20, 1955, and the second in Washington, D. C., on March 22, 1956.

There have been two resignations from the committee during the year. At present the committee is composed of 37 members, of whom 11 are classified as producers, 16 as consumers, and 10 as general interest members.

The officers and Advisory Committee members elected for the ensuing term of two years are as follows:

Chairman, J. M. Clark.

Vice-Chairman, F. G. Church.

Secretary, C. F. Graham.

Advisory Committee: O. W. Chandler, C. O. Durbin, V. O. Hatch, G. Kaufman, R. E. Vogel.

Unanimous approval was given at the March meeting to a tribute of appreciation for H. R. Wolf the retiring charter chairman. The citation to be presented Mr. Wolf in the form of a scroll recognizes his outstanding leadership as chairman. His wise counsel, his scientific integrity, the high regard of his friends and associates on the committee, have provided the inspiration and guidance for the promotion of knowledge of antifreeze properties and the development of test methods since the founding of the committee.

The Board of Directors, at a special presentation by President C. H. Fellows, on April 25, 1956, at Detroit, Mich., conferred upon H. R. Wolf the ASTM Award of Merit. This Award was made in recognition of longtime, valued service to the Society, especially supporting standardization and research work in Committee D-2 on Petroleum Products and Lubricants, and one of the organizers of Committee D-15 on Engine Antifreezes of which he served as chairman from 1947 to 1956.

TENTATIVES CONTINUED WITHOUT REVISION

Committee D-15 recommends that the following tentative specification and methods be continued as tentative without revision:

Tentative Specification for:

Hydrometer Thermometer Field Test for Engine Antifreezes (D 1124 – 53 T).

Tentative Methods of Test for:

Ash Content of Concentrated Engine Antifreezes (D 1119 – 53 T), and pH of Concentrated Engine Antifreezes (D 1278 – 53 T).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Antifreeze Field Testers (F. G. Church, chairman) is revising the Tentative Specification for the Hydrometer-Thermometer Field

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*** Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.**

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Tester for Engine Antifreezes (D 1124 - 53 T) to include a test method for obtaining basic data with hydrometers for the initial calibration of these testers for primary use by antifreeze producer and tester manufacturers. Collaborative tests are in progress to establish the accuracy and adequacy of the test method. A study group on Direct-Reading Testers (J. J. Ondrejcin, chairman) is continuing its investigation of other testers such as the direct-freeze type in which a sample of the antifreeze is frozen with dry ice or expanding CO_2 gas.

Subcommittee IV on Chemical Properties (E. G. Travis, chairman).—The Study Group on Karl Fischer Moisture Method reported the results of laboratory investigation of inhibitor interference with Karl Fischer reagent. Due to the complexity of the problem, the study group chose to discontinue the laboratory work, and to add a significance statement to the present method pointing out to users of the method the possibility of interference of Karl Fischer's reagent by components of antifreeze.

In addition, the study group has recommended changing the present moisture determination method to incorporate the new glycol ether-base reagent as opposed to the older methanol-base reagent.

Subcommittee V on Effect of Antifreeze on Rubber Hose (O. W. Chandler, chairman).—Collaborative test results on a proposed glassware test for determining the effect of engine antifreezes on cooling system hoses indicate that the proposed method is satisfactory. It has been agreed that an appendix should be added to the method to explain fully the test significance as well as to provide a detailed discussion, illustrated by actual test results, of test accuracy and precision.

Subcommittee VI on Simulated and Actual Service Testing (C. H. Sweatt, chairman).—The Study Group on Glassware Test Methods (E. L. Gibson, chairman) is developing a glassware screening test to evaluate the foaming properties of engine antifreezes.

The newly organized Study Group on Service Simulation Test Methods, with Mr. V. O. Hatch as acting chairman, is proceeding with the development of a circulating type corrosion and foaming test to simulate conditions encountered in service. Collaborative tests will be initiated as soon as design details are completed.

Subcommittee VII on Specifications (J. M. Clark, chairman) was inactive during the year; it is awaiting the completion of Projects in Subcommittees IV and VI.

This report has been submitted to letter ballot to the committee, which consists of 37 voting members; 37 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

H. R. WOLF,
Chairman.

C. F. GRAHAM,
Secretary.

REPORT OF COMMITTEE D-16

ON

INDUSTRIAL AROMATIC HYDROCARBONS AND RELATED MATERIALS*

Committee D-16 on Industrial Aromatic Hydrocarbons and Related Materials and its subcommittees held meetings on June 27 and 28, 1955, in Atlantic City, N. J., and on February 27 and 28, 1956, in Buffalo, N. Y.

A new Subcommittee H on Alkyl Aromatics for Detergent Intermediates was formed with S. S. Kurtz, Jr., as chairman.

The present officers were re-elected for the ensuing term of two years.

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption the following revision of the Standard Method of Test for Distillation of Industrial Aromatic Hydrocarbons (**D 850 – 55**)[1] and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revision may be referred to letter ballot of the Society.

Section 2 (*f*).—Change to read as follows:

> *Heater*.—An electric heater or a bunsen burner, fully adjustable and capable of giving sufficient heat to distill the product at the required rate. When a bunsen burner is used, the burner shall be adjusted so as to produce an entirely blue flame. In case of dispute concerning results obtained with gas heat *versus* electric heat, gas heat shall be accepted as the standard.

This recommendation has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee A on Monocyclic Aromatics (S. S. Kurtz, Jr., chairman) has prepared a revision of Specification for Industrial 90 Benzene (D 837).[3] A method for the determination of thiophene in benzene, to include values of less than 1 ppm, will be recommended for publication as information only[4] since the method will probably be extensively revised before being considered for publication as tentative next year. Current projects include investigation of spectroscopic methods for determining isomer distribution in nitration grade xylene, the coulometric procedure for determining the bromine index of benzene, and revision of the existing potentiometric method.

Subcommittee B on Polycyclic Aromatics (M. Mitchell, chairman) has reached agreement on a method for determining color of refined naphthalene. Further work on methods for solidification point and acid wash test is planned.

Subcommittee C on Phenolic Compounds (V. S. Morello, chairman) has conducted cooperative tests on a method for determining water content of refined phenol with excellent results. Progress was made on methods for solidification point and water solubility.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] 1955 Book of ASTM Standards, Part 5.

[2] The letter ballot vote on this recommendation was favorable; the results of the vote are on record at ASTM Headquarters.

[3] See Editorial Note.

[4] This method appears in the 1956 Compilation of ASTM Standards on Benzene, Toluene, Xylene, Solvent Naphtha.

Subcommittee D on Nitrogen Heterocyclics (W. Sisco, chairman) is conducting cooperative tests on methods for determining boiling range and water content of refined pyridine and refined quinoline. A solidification point method for quinoline is also being studied.

Subcommittee E on Thermometry (K. H. Ferber, chairman) is working on the development of a series of solidification point thermometers, graduated in 0.1 C, 30 deg range, 76 mm immersion. Contact with Subcommittee 17 on Thermometers of Committee E-1 on Methods of Testing is being maintained with the aim of securing official listing of the series as ASTM thermometers.

Subcommittee G on Statistical Procedure (W. J. Tancig, chairman) is functioning as a consultant body to aid in the cooperative programs of the other subcommittees.

This report has been submitted to letter ballot of the committee, which consists of 56 voting members; 37 members returned their ballots, of whom 34 have voted affirmatively, and 0 negatively.

Respectfully submitted on behalf of the committee,

D. F. GOULD,
Chairman.

F. J. POWELL,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-16 presented to the Society through the Administrative Committee on Standards the recommendation that the Standard Specifications for Industrial 90 Benzene (D 837 – 50) be revised and reverted to tentative status. This recommendation was accepted by the Standards Committee on August 9, 1956, and the tentative specifications appear in the 1956 Supplement to Book of ASTM Standards, Part 5.

REPORT OF COMMITTEE D-17

ON

NAVAL STORES*

Committee D-17 on Naval Stores held one meeting during the year, on June 28, 1955, at Atlantic City, N. J.

The committee consists of 38 members, of whom 17 are classified as producers, 10 as consumers, and 11 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, V. E. Grotlisch.
Vice-Chairman, J. L. Boyer.
Secretary, W. A. Kirklin.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1955 Annual Meeting, Committee D-17 presented to the Society through the Administrative Committee on Standards the recommendation that the Standard Methods of Testing Tall Oil (D 803 - 51) be revised and reverted to tentative status. The revised tentative methods appear in the 1955 Book of ASTM Standards, Part 4.

ADOPTION OF TENTATIVE AS STANDARD WITHOUT CHANGE

The committee recommends that the Tentative Methods of Test for Unsaponifiable Matter in Rosin (D 1065 - 54 T)[1] be approved for reference to letter ballot of the Society for adoption as standard without change.

The recommendation in this report has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Softening Point of Rosin (J. M. Schantz, chairman).—More than 20 years have elapsed since the modified method of test for softening point by ring and ball method (now Method E 28 - 51 T) was published by Committee D-17 (see appendix to 1933 Annual Report). Although this method is still important in both routine and referee testing of the softening point of rosins and related naval stores products, and referred to in most specifications for this class of material, the committee will begin collaborative study on the so-called Hercules drop method as an additional method for testing softening point of rosin. This method will be discussed at the Annual Meeting.

Subcommittee II on Crystallization of Rosin (S. R. Snider, chairman).—Samples of gum rosin representing all areas of production, and made from pine gum collected both in summer and late fall, were tested in accordance with the proposed method for determining the so-called crystallization tendency of rosin published as Appendix I to the 1955 Annual Report of Committee D-17.[3] The results appeared to confirm an earlier assumption that the tendency for gum rosin to crystallize is in some manner related to the geographical location of the timber stands from which the crude gum or oleoresin was collected. A collaborative test program will be based upon this study.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17 - 22, 1956.

[1] 1955 Book of ASTM Standards, Part 4.

[2] The letter ballot vote on this recommendation was favorable; the results of the vote are on record at ASTM Headquarters.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 55, p. 531 (1955).

Subcommittee IV on Chemical Analysis of Rosin (R. Herrlinger, chairman).—Samples of mixtures of fatty and rosin acids of known composition were prepared for use in collaborative work toward a more reliable method for determining fatty acids in rosin. Such a test has particular importance in the analysis of tall-oil rosin, in which fatty acids are usually present but must be closely controlled and held to a low value. Efforts to prepare known mixtures by combining pure abietic acid with pure fatty acids were not successful because, when such a mixture cools and solidifies, the abietic acid tends to crystallize, thereby "squeezing" out the fatty acid from the mixture to produce a nonuniform mass not fully suitable as an authentic sample on which to carry out the proposed collaborative work. This difficulty was not encountered when the fatty acids were combined with refined wood rosins. It is planned to include both the modified Wolfe method, as described in the tall-oil test methods, and the Linder-Persson method, which has been recommended. Lack of the necessary laboratory equipment and insufficient interest has postponed the study of spectrophotometric procedures used to identify and quantitatively determine the rosin acids present in rosins.

Subcommittee VII on Terpene Hydrocarbons and Pine Oil (R. E. Price, chairman).—The directions for conducting the distillation test on turpentine, dipentene, and pine oil, as given in the several standard methods of test for these materials, have been reviewed with emphasis on the corrections applied to the observed initial distilling temperature and the temperature when the distillation is stopped, and to adjust the test for variations in atmospheric pressure. It was found that the temperature correction now used for turpentine, 0.057 C per mm of mercury variation from standard pressure of 760 mm at 0 C, was determined and published over 100 years ago by the French chemist, Regnault. This value is not in agreement with the correction computed on the basis of the distilling range for turpentine by the Sydney Young equation, which is now generally accepted as being applicable to any distillation test. The Sydney Young value for turpentines is 0.052 C per mm of mercury, while for dipentene and pine oils the values are 0.054 and 0.059, respectively. Editorial revisions based on this work are being prepared for inclusion in the dipentene Method D 801 and pine oil Method D 802.

The directions for performing the test for unpolymerized residue in turpentine have been reviewed in view of the fact that some difficulty has been reported in using the ASTM Standard Methods of Sampling and Testing Turpentine (D 233). High results in excess of the 2.0 per cent permitted in the standard specifications for turpentine were reported by several laboratories, most often on steam-distilled wood turpentines, but with referee testing the polymerization residues were usually found to be within the specification limits.

This report has been submitted to letter ballot of the committee, which consists of 38 voting members; 26 members returned their ballots, of whom 24 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

V. E. GROTLISCH,
Chairman.

W. A. KIRKLIN,
Secretary.

REPORT OF COMMITTEE D-18

ON

SOILS FOR ENGINEERING PURPOSES*

Committee D-18 on Soils for Engineering Purposes held its Annual Meeting at Atlantic City, N. J., on June 29, 1955. Executive Subcommittee meetings were held on June 27, 1955, at Atlantic City and on January 16, 1956, at Washington, D. C.

A Symposium on Judgment Factors in Soil Testing[1] was held during the 1955 Annual Meeting to provide discussions on past, present, and future activities of the committee with respect to standardizing certain soil tests. It is believed the discussions have done much to clear the way for future constructive work by the committee.

A soils session also was held during the 1955 Annual Meeting consisting of five papers on general soils subjects; a similar session is planned at the 1956 Annual Meeting as well as a Symposium on Vane Shear Testing.[2]

During the 1955 Annual Meeting the second Hogentogler Memorial Award of Committee D-18 was presented to Messrs. H. G. Mason, J. A. Bishop, L A. Palmer, and P. O. Brown for their paper on "Piles Subjected to Lateral Thrust." On June 28, 1955, Committee Chairman E. J. Kilcawley received the Society Award of Merit for continuous and outstanding service to the Society through Committee D-18 activities.

Soils sessions are scheduled during the Second Pacific Area National Meeting of the Society (September 16 to 22, 1956) in Los Angeles, Calif., and 15 papers have been secured.[3] Responsibility for the sessions rests with a special committee consisting of F. J. Converse (chairman), W. G. Holtz, and R. Horonjeff.

A special committee to consider revisions of the procedures for soil testing was formed consisting of the subcommittee chairmen, with W. J. Turnbull, chairman. The summer of 1957 has been set as the deadline for the completion of the work.

A review of the form of ASTM standards for soils is to be made by a special committee consisting of the subcommittee chairmen and R. F. Leggett, with W. G. Holtz, chairman. The committee desires definite suggestions on how the standards might be improved, taking into account the peculiarities of standardizing soil tests.

A. E. Cummings, chairman of Subcommittee R-11, and D. W. Taylor, committee member, died during the past year. Both of these men have been active in committee work for many years and will be greatly missed by their colleagues.

N. W. McLeod has been appointed chairman of Subcommittee R-4 on Physical Properties of Soils, and W. S. Housel has been appointed chairman of Subcommittee R-11 on Pile Load Bearing Tests.

The committee consists of 129 individual members and 19 consultants. There are 99 voting memberships divided as follows: producers, 21; consumers, 37; and general interest, 41.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] See Donald M. Burmister, "Judgment and Environment Factors in Soil Investigations," ASTM Bulletin No. 217, October, 1956, p. 55 (TP 189).

[2] Issued as separate publication *ASTM STP No. 193.*

[3] Issued as separate publication *ASTM STP No. 206.*

NEW TENTATIVES

The committee recommends that the following method be accepted for publication as tentative as appended hereto:[4]

Method of Testing Graded Aggregate Road Mixes for Certain Water Soluble Chlorides Present as Admixtures. (Jointly with Committee D-4 on Road and Paving Materials; see D-4 Report for copy of the method.)

REVISION OF TENTATIVE

The committee recommends that the Tentative Method of Test for Moisture-Density Relations of Soils (**D 698 – 42 T**)[5] be revised as appended hereto[4] (contingent on similar action by Committee D-4).

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following tentatives which have stood for two or more years be continued without change:

Tentative Method of:

Grain-Size Analysis of Soils (**D 422 – 54 T**),
Test for Liquid Limit of Soils (**D 423 – 54 T**),
Test for Plastic Limit and Plasticity Index of Soils (**D 424 – 54 T**),
Testing Soil-Bituminous Mixtures (**D 915 – 47 T**),
Test for Shear Strength of Flexible Road Surfaces, Subgrades and Fills by the Burgraff Shear Apparatus (**D 916 – 47 T**),
Test for Bearing Capacity of Soil for Static Load on Spread Footings (**D 1194 – 52 T**),
Repetitive Static Load Tests of Soils, for Use in Evaluation and Design of Airport and Highway Pavement (**D 1195 – 52 T**),
Nonrepetitive Static Load Tests of Soils, for Use in Evaluation and Design of Airport and Highway Pavements (**D 1196 – 52 T**), and
Test for Load-Settlement Relationship for Individual Piles (**D 1143 – 50 T**).

Tentative Definitions of:

Terms and Symbols Relating to Soil Mechanics (**D 653 – 42 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[6]

SUBCOMMITTEE ACTIVITIES

Subcommittee G-1, Editorial (E. E. Bauer, chairman) reviewed the revision of the Tentative Method of Test for Moisture-Density Relations of Soils (D 698) referred to earlier in the report.

Subcommittee G-2 on Special Papers (Paul Phelan, chairman) arranged for the presentation of five papers at a general soils session to be held during the 1956 Annual Meeting of the Society.

Subcommittee G-3 on Nomenclature and Definitions (C. R. Foster, chairman) has completed the review of approximately 600 terms relating to soils mechanics and foundation engineering and plans to recommend their incorporation in the Definitions of Terms and Symbols Relating to Soil Mechanics (D 653 – 42 T).

Subcommittee R-2 on Sampling and Related Field Testing for Soil Investigation (J. O. Osterberg, chairman) prepared the three proposed methods for auger sampling, thin wall tube sampling, and penetration testing and splin spoon sampling, referred to earlier in the report. The subcommittee has arranged for several papers to be presented at a Symposium on Vane Shear Testing[2] during the 1956 Annual Meeting of the Society.

Subcommittee R-3 on Physical Characteristics of Soils (E. E. Bauer, chairman) prepared the revisions of the Tentative Method of Test for Moisture-Density Relations of Soils (D 698) referred to earlier in the report.

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[4] The new and revised tentatives appear in the 1956 Supplement to Book of ASTM Standards, Part 3.

[5] 1955 Book of ASTM Standards, Part 3.

[6] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Section D is sponsoring a cooperative testing program to determine the best procedure to be used for maximum and minimum void ratio values required for relative density determinations. A survey is being conducted to determine the use being made of the Method of Test for Centrifuge Moisture Equivalent of Soils (D 425 – 39), and Method of Test for Field Moisture Equivalent of Soils (D 426 – 39). Consideration is being given to suggestions for revisions of the Method of Grain-Size Analysis of Soils (D 422 – 54 T).

Subcommittee R-5 on Structural Properties of Soils (D. M. Burmister, chairman) is considering which structural property tests might be standardized in view of the necessity to consider environmental influences in interpreting the test results. Section C plans to work on a method for unconfined compression testing.

Subcommittee R-7 on Identification and Classification of Soils (A. A. Wagner, chairman) is reviewing the number of classification systems to be included in the revised edition of the Procedures for Testing Soils.

Subcommittee R-9 on Dynamic Properties of Soils (R. K. Bernhard, chairman). —The Bibliography on Soil Dynamics is being brought up-to-date, and a discussion of some current dynamic research projects is planned for the next meeting. A glossary of terms and definitions relating to soil dynamics has been referred to Subcommittee G-3.

Subcommittee R-11 on Pile Load Bearing Tests (W. S. Housel, chairman) is considering revisions of the Method of Test for Load-Settlement Relationship for Individual Piles (D 1143 – 50 T).

This report has been submitted to letter ballot of the committee, which consists of 100 voting members; 65 members returned their ballots all of whom voted affirmatively.

Respectfully submitted on behalf of the committee,

E. J. KILCAWLEY,
Chairman.

W. G. HOLTZ,
Secretary.

REPORT OF COMMITTEE D-19
ON
INDUSTRIAL WATER*

Committee D-19 on Industrial Water and its subdivisions held meetings on June 29 to July 1, 1955, in Atlantic City, N. J., and on January 23 to 25, 1956, in Biloxi, Miss.

A Symposium on High-Purity Water Corrosion, sponsored by the committee at the 1955 Annual Meeting of the Society, has been published as *ASTM STP 179*.

A Symposium on Industrial Water[1], consisting of the papers listed below, will be sponsored by the committee during the Second Pacific Area National Meeting of the Society (September 16–22, 1956) at Los Angeles, Calif.:

"Industrial Waste Problems in Southern California," by T. C. Wilson, Los Angeles Bureau of Standards.

"Water Pollution Control in the Los Angeles Area," by C. B. Johnston, Los Angeles Regional Water Pollution Control Board No. 4.

"Sea Water Purification," by O. M. Elliott, Sun Oil Co.

"Committee D-19: The First Quarter Century," by R. C. Adams, U. S. Naval Engineering Experiment Station.

"The Use of Organic Flocculants and Flocculating Aids in the Treatment of Industrial Water and Industrial Waste Water," by J. K. Rice, Cyrus Wm. Rice & Co.

This symposium has been arranged by L. Drew Betz, Chairman of Subcommittee VII on Industrial Waste Water.

Committee D-19 is very much interested in the Symposium on Steam Quality[2] which is being sponsored by the Joint Research Committee on Boiler Feedwater Studies at the 1956 Annual Meeting of the Society. The following papers comprise the symposium:

"Measurement and Purification of Steam to 0.01 ppm Total Dissolved Solids," by W. B. Gurney, Gulf States Utilities Co.

"Steam Purity Determination by Tracer Techniques," by Earl E. Coulter, the Babcock & Wilcox Co.

"Comments on Corrections to Steam Conductivity Measurements," by R. O. Parker, Griscom-Russell Co.

"Construction and Operation of Larson-Lane Steam Purity and Condensate Analyzers," by A. B. Sisson, Commonwealth Edison Co., F. G. Straub, University of Illinois, and R. W. Lane, Illinois State Water Survey.

The committee is continuing preparation of a new edition of the Manual on Industrial Water as discussed in detail in the report of Subcommittee II.

Committee D-19 consists of 131 members; they are not classified.

As of July 2, 1955, there were 70 task groups of the committee allocated among the Advisory Committee and the six technical subcommittees.

The printed Guide for the Administration and Operation of Committee D-19 on Industrial Water was distributed to the members in April, 1956. The edition includes Part I, "Regulations Governing the Committee;" Part II, "Administration and Operation;" and Part III, "Recommended Form of Committee D-19 Methods and Specifications."

*** Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.**

[1] Issued as a separate publication *ASTM STP No. 207.*

[2] Issued as a separate publication *ASTM STP No. 192.*

Two of the committee members were honored in 1955. They are Robert C. Adams, vice chairman of the committee, the recipient of the ASTM Award of Merit, and Everett P. Partridge, the recipient of the 1955 Max Hecht Award.

John E. Kinney, Consulting Sanitary Engineer, and Claude K. Rice, of the Gulf Oil Corp., were appointed as the Society representatives to serve on the newly organized Joint Committee on Uniformity of Methods of Water Examination.[3] With the organization of this joint committee, the Committee D-19 Informal Task Group on Uniformity of Methods was discharged.

The officers elected by virtue of the letter vote on this report, to serve for the two-year term starting June 22, 1956, are as follows:

Chairman, Max Hecht.
Vice Chairman, Robert C. Adams.
Vice Chairman, Fred R. Owens.
Secretary, Orrin M. Elliott.

Committee members elected to serve on Subcommittee I Advisory for the three-year term starting June 22, 1956, are as follows:

Robert E. Price.
J. F. J. Thomas.

PROPOSED METHODS TO BE PUBLISHED AS INFORMATION

The committee recommends for publication as information the Proposed Method of Test for Appearance Properties of Industrial Water and Industrial Waste Water, as appended hereto.[4]

NEW TENTATIVES

The committee recommends that the following four methods be accepted for publication as tentative, as appended hereto:[5]

Methods of Test for:

Ammonia in Industrial Water and Industrial Waste Water,
Sodium and Potassium Ions in Industrial Water and Water-formed Deposits by Flame Photometry,
Specific Gravity of Industrial Water and Industrial Waste Water, and
Residual Chlorine in Industrial Waste Water.

Supporting data for the Method of Test for Ammonia is given in the Appendix to this report.[6]

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following two tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Specifications for Reagent Water (**D 1193 - 55 T**),[7] and
Tentative Methods of Test for Oily Matter in Industrial Waste Water (**D 1340 - 54 T**).[7]

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption the following revisions of Standard Definitions of Terms Relating to Industrial Water and Industrial Waste Water (**D 1129 - 55**)[7] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Add the following new definitions:

Precision.—The degree of agreement of repeated measurements of the same property, expressed in terms of dispersion of test results about the arithmetical mean result obtained by repetitive testing of a homogeneous sample under specified conditions. The precision of a method is expressed quantitatively as the standard deviation computed from the results of a series of controlled determinations.

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[3] See ASTM BULLETIN, July, 1956, p. 44.

[4] Published in Third Printing of the "Manual on Industrial Water," (1957). Issued as separate publication *ASTM STP No. 148B.*

[5] The new tentatives appear in the 1956 Supplement to Book of ASTM Standards, Part 7

[6] See p. 355.

[7] 1955 Book of ASTM Standards, Part 7.

Specific Gravity.—The ratio of the weight in air of a given volume of the sample to the weight in air of an equal volume of reagent water, both being determined at the standard reference temperature of 15.6 C (60 F). It is expressed as follows:

Specific Gravity, 15.6/15.6 C (60/60 F) . . .

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that all tentative methods and specifications under its jurisdiction be continued as tentative with the exception of those recommended for adoption as mentioned earlier in the report.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[8]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I, Advisory (Max Hecht, chairman).—The over-all activities of the subcommittee included coordination of methods production, approval of the Manual project (covered under Subcommittee II), approval of the symposium for the Second Pacific Area National Meeting, and the implementation of the final phase of the committee reorganization which was effective July 2, 1955.

The recommendations set forth previously in this report indicate the activity of the committee during the current year on methods production. Sixty-six of the task groups of the committee are engaged in studies for the preparation of proposed methods and of proposed revisions of published methods. As of June, 1955, a total of 50 methods and specifications prepared by the committee had been approved by the Society for publication.[7]

Subcommittee II on Definitions and General Specifications (S. K. Love, chairman).—The integrated outline of the nine chapters of the revised edition of the Manual has been approved by Committee D-19. It is expected that the authors will begin writing their assigned chapters in the very near future. The chapter titles and the designated authors are as follows:

Foreword and Introduction (Richard D. Hoak, Mellon Institute).
Chapter I. Uses of Industrial Water (James K. Rice, C. W. Rice Co.).
Chapter II. Effects of Impurities on Water Uses (Roy F. Weston, Weston, Eckenfelder and Associates).
Chapter III. Self-Purification of Streams (George D. Beal, and William W. Leathen, Mellon Inst.).
Chapter IV. Composition of Industrial Water and Water-formed Deposits (S. K. Love, U. S. Geological Survey).
Chapter V. Treatment of Industrial Water and Industrial Waste Water (Frank N. Alquist, Dow Chemical Co., and Bruce W. Dickerson, Hercules Powder Co.).
Chapter VI. Sampling Industrial Water and Waste Water (J. E. Kinney, Consultant).
Chapter VII. Analysis of Industrial Water and Waste Water (James M. Seamon, National Aluminate Co., and W. Allan Moore, U. S. Public Health Service).
Chapter VIII. Sampling and Identification of Water-formed Deposits (R. K. Scott, Harbison Walker Refractories Co.).
Chapter IX. Analysis of Water-formed Deposits (Frank E. Clarke, U. S. Naval Engineering Experiment Station).
Appendix (Dudley K. French, Consultant).
Glossary (Richard D. Hoak, Mellon Institute).
Index (Richard D. Hoak, Mellon Institute).

Definitions were approved for precision and specific gravity, as mentioned earlier in the report. A definition for accuracy was retained in the task group for further study, and definition for sensitivity is under consideration.

The Specifications for Reagent Water (D 1193) were recommended for adoption as standard as mentioned earlier in the report.

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[8] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

The Task Group on Scheme for Analysis was transferred from Subcommittee IV.

Subcommittee III on Methods of Sampling of Industrial Water (E.F.Davidson, chairman) is studying the Tentative Method of Sampling Steam (D 1066 – 54 T) and the Tentative Specifications for Equipment for Sampling Industrial Water and Steam (D 1192 – 51 T) with regard to new methods and equipment which may be required for supercritical pressure operation and monotube systems. The committee has been requested to study also subatmospheric sampling, especially of small, low-pressure, heating plant boilers.

Some progress has been made on formulating a program for studying radioactive materials in industrial water and industrial waste water. The specific function of Subcommittee III will be the study of problems in connection with sampling methods and techniques.

Subcommittee IV on Methods of Analysis of Industrial Water (F. E. Clarke, chairman) prepared methods for hydrazine, silica, acidity and basicity, iron, fluoride, chloride, phosphate, and sulfate, the first three methods being prepared jointly with Subcommittee VII.

Final drafts of new methods were approved for ammonia, referred to earlier in the report, and for micro silica. The latter will be combined with the present silica method (D 859).

Preliminary drafts or other important preliminary work have been completed on methods for micro iron, high nitrite, copper, zinc, referee sulfide, nickel, lead, fluoride, low sulfate, volatile amines, sulfite, and hydrazine.

Subcommittee V on Methods of Testing (A. O. Walker, chairman) is recommending that the Tests for Electrical Conductivity of Industrial Water (D 1125) and the Method for Identification of Types of Microorganisms in Industrial Water (D 1128) be retained as tentative since these methods are in process of revision.

The work on the final draft of the Proposed Method for Sodium and Potassium Ions in Industrial Water and Water-formed Deposits, referred to previously in the report, was completed at the January meeting.

A proposed method for the determination of gaseous hydrogen in industrial water is nearing completion.

A statistical review of the results of the first round-robin test on surface tension gave inconclusive results. A revised test program, based on more sound statistical foundation was reviewed and adopted.

A draft of the spectroscope method of analysis will be submitted to the task group for comment at the June meeting.

Subcommittee VI on Corrosion and Performance Testing (F. N. Alquist, chairman).—During the past year the task group on ion exchange has been working on methods for measuring cation exchange capacity. Other problems under study are resistance to aggressive attack, anion exchange capacity, and operating capacity.

A task group, after two years study and limited field testing, has prepared a revised draft of a method of testing corrosion and deposits in tubular heat exchange goods. This method will be experimentally checked before the next meeting.

A temporary task group, formed at the June meeting in 1954, recommended that Subcommittees III, IV, V, VI and VII cooperate in order to coordinate methods to be used to measure radioisotopes in industrial water. This proposal was approved.

The task group on radioactive tracers

recommended the following course of action to Committee D-19:

1. A task group of Subcommittee V should be created to prepare methods for:
    - (*a*) The determination of gamma radiation from sodium-24 or potassium-42 in a sample of water where the concentration range is from 1×10^{-5} microcuries per ml to 1×10^{-2} microcuries per ml.
    - (*b*) The determination of beta radiation from phosphorus-32 in a sample of water where the concentration range is from 1×10^{-5} microcuries per ml to 1×10^{-2} microcuries per ml.
2. A task group of Subcommittee IV should be created to prepare methods for separating sodium-24, potassium-42, or phosphorus-32 from water samples by ion exchange or chemical precipitation to achieve a 100 to 1000-fold concentration of these substances over their 1×10^{-8} to 1×10^{-5} microcuries per ml concentration in the sample.
3. The task group on radioactive tracers should attempt to prepare a method on the use of phosphorus-32 in steam purity determinations and a method for the use of sodium-24 or potassium-42 in testing the hydraulic characteristics of gravity separators.
4. A chairman for the task group on radioactive tracers should be selected from among those whose work involves the use of radiation measuring instruments or the handling of radioactive materials.
5. The task group chairmen of such task groups concerned with radioactive material, as may be created in Subcommittees III, IV, VI, and VII, should be members of the task group on radiation measurement technique to be created in Subcommittee V.

Subcommittee VII on Industrial Waste Water (L. D. Betz, chairman).—There are seventeen active task groups working in this subcommittee, several of which are joint task groups with Subcommittee IV.

The subcommittee recommended that the Method of Test for Oily Matter in Industrial Waste Water (D 1340) be adopted as standard. Three other methods, for Chlorine Requirements (D 1291), for Odor (D 1292), and for Toxicity to Aquatic Life (D 1345), were recommended to be continued as tentative as reported earlier in this report. The Subcommittee prepared the proposed Tentative Methods of Test for Specific Gravity of Industrial Water and Industrial Waste Water referred to earlier in the report.

The task group on acidity, basicity and pH have reviewed the revised method (D 1067) and have suggested several minor changes all of which are editorial in nature.

The task group on oxygen demand, after considerable discussion, is now ready to proceed with the preliminary work on the B.O.D. test.

The task group on taste and odor is checking into the work done by Kinney and Hoak on threshold odors in relation to phenol and the development of phenol odors from natural sources.

The Task Group on Chlorine Residual has prepared the method for this determination referred to earlier in the report.

The task group on oily matter has agreed that infrared and irridescence cannot be used as a means for determining oily matter. It has, however, suggested that Subcommittee V consider irridescence as a means for measuring appearance properties.

The task group on phenolic compounds

has circulated a preliminary draft of a procedure for determining phenol using 4-aminoantipyrine for comments and suggestions.

The task group on sampling is studying methods of sampling industrial waste containing floating materials, and the task group on gaging is working on methods of flow measurement and specifications for equipment for flow measurement.

The task group on reporting has agreed that since practically all state, interstate, and federal water pollution control agencies require reports in parts per million or parts per billion, industry should adopt the same system of reporting, except for certain in-plant operations.

Other task groups on sulfur compounds, cyanides, organic nitrogen, chromium, and detergents are diligently working on the development of methods for the determination of these substances in industrial waste water.

This report has been submitted to letter ballot of the committee, which consists of 130 members; 100 members returned their ballots, of whom 85 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

MAX HECHT,
Chairman.

ORRIN M. ELLIOTT,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-19 presented to the Society through the Administrative Committee on Standards a recommendation for the revision of the Tentative Methods of Test for Sodium and Potassium Ions in Industrial Water and Water-formed Deposits by Flame Photometry (D 1428 – 56 T). This recommendation was accepted by the Standards Committee on September 5, 1956, and the revised methods appear in the 1956 Supplement to Book of ASTM Standards, Part 7.

APPENDIX

SUPPORTING DATA RELATING TO THE DETERMINATION OF AMMONIA IN INDUSTRIAL WATER AND INDUSTRIAL WASTE WATER[1]

In Table I, the recoveries of ammonia obtained at the various pH levels are given. An examination of the data presented in this table shows several pertinent facts related to the recovery of ammonia. It will be noted that at low ammonia concentrations (0.1 mg.) the percentage recovery increases with increasing pH. At pH 7.4 only 75 per cent of the ammonia added is accounted for, and after the first 200 ml of distillate no more ammonia is detectable. This is no doubt due to a tailing off of ammonia in subsequent 50-ml portions in concentrations too low to detect. In a subsequent distillation at a 0.1 mg concentration where 300 ml of distillate was collected and an aliquot nesslerized, a recovery of 99 per cent of the ammonia was obtained. Therefore, it is preferable to collect an adequate volume of distillate and nesslerize an aliquot instead of individual 50-ml portions. Another fact that the data show is that the rate of ammonia distillation is slower at a pH of 7.4 than at higher pH values.

TABLE I.—AMMONIA RECOVERY IN MILLIGRAMS.

50-ml Distilled Portion No.	0.1 mg NH_3 as N_2 at pH				1.0 mg NH_3 as N_2 at pH				10 mg NH_3 as N_2 at pH			
	7.4	9.0	11.0	14	7.4	9.0	11.0	14	7.4	9.0	11.0	14
1....	0.036	0.056	0.056	0.060	0.58	0.76	0.74	0.66	5.01	6.97	7.30	7.00
2....	0.023	0.0165	0.017	0.040	0.26	0.22	0.245	0.27	2.33	1.93	1.71	1.93
3....	0.010	0.0065	0.007	0.007	0.12	0.052	0.048	0.076	1.20	0.55	0.43	0.70
4....	0.006	0	0.004	0	0.058	0.016	0.024	0.021	0.54	0.135	0.106	0.145
5....	0	0	0	0	0.025	0.0045	0.005	0.008	0.28	0.030	0.025	0.034
6....	0	0	0	0	0.005	0	0	0	0.1125	0.008	0	0.085
7....	0	0	...	...	0.004	0	0	0	0.054	0	0	0
8....	0	...	...	...	0	...	...	...	0.0195	0	0	0
9....	...	...	...	...	...	...	...	...	0.005	...	...	...
10....	...	...	...	...	...	...	...	...	0	...	...	...
Total	0.075	0.079	0.084	0.107	1.052	1.053	1.062	1.035	9.55	9.62	9.57	9.81

The main reason for the acceptance of a pH of 7.4 has been to minimize the hydrolysis of organic nitrogen compounds. Such hydrolysis would result in an increase in the amount of ammonia found in a given sample. The effect of pH on the amount of ammonia recoverable from typical organic nitrogen compounds that might occur simultaneously with ammonia in a given sample was studied. The results obtained are shown in Table II.

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[1] These data were obtained at the Robert A. Taft Sanitary Engineering Center, U. S. Public Health Service, Cincinnati, Ohio.

Glycine, the simplest of the amino acids, gave a recoverable ammonia nitrogen of 0.018 mg out of a total of 10 mg at all pH values. However, the glycine used was not ammonia-free and by direct nesslerization 0.5 mg of ammonia nitrogen was obtained. It can be concluded, therefore, that in the case of glycine no hydrolysis took place regardless of the pH used. Glutamic acid also showed no hydrolysis of the amino-nitrogen. The data for acetamide shows no hydrolysis at pH 7.4 and increasing hydrolysis with subsequent increase in pH. It will be noted that at pH 14.0, 97 per cent of the acetamide was recoverable as ammonia nitrogen. Urea, which has been considered easily hydrolyzable, did not give the recoverable ammonia concentrations expected. At pH 7.4 only 7.7 per cent of recoverable ammonia was obtained and this increased to only 17 per cent at pH 14.0.

TABLE II.—HYDROLYSIS OF ORGANIC NITROGEN COMPOUNDS.

50-ml Distilled Portion No.	Glycine 10 mg as N_2 at pH				Glutamic Acid 10 mg as N_2 at pH				Acetamide 10 mg as N_2 at pH				Urea 20 mg as N_2 at pH			
	7.4	9.0	11.0	14	7.4	9.0	11.0	14	7.4	9.0	11.0	14	7.4	9.0	11.0	14
1....	0.008	0.012	0.012	0.014	0	0	0	0	0	0.0085	0.015	5.82	0.065	0.14	0.85	0.205
2....	0.006	0.006	0.006	0.004	0	0	0	0	0	0.0135	0.030	2.76	0.14	0.17	0.135	0.32
3....	0.004	0	0	0	0	0	0	0	0	0.0155	0.032	0.84	0.15	0.20	0.165	0.34
4....	0	0	0	0	0	0	0	0	0	0.016	0.034	0.205	0.24	0.21	0.200	0.38
5....	0	0	0	0	0	0	0	0	0	0.0165	0.036	0.042	0.26	0.20	0.220	0.40
6....	0	0	0	0	0	0	0	0	0	0.0165	0.040	0.010	0.21	0.21	0.240	0.46
7....	0	0	0	0	0	0	0	0	0	0.0195	0.044	0	0.21	0.22	0.210	0.50
8....	0	0	0	0	0	0	0	0	0	0.020	0.050	0	0.26	0.22	0.220	0.80
9....	...	...	...	...	...	...	...	...	0	...	...	0	...	...	...	...
Total	0.018	0.018	0.018	0.018	0	0	0	0	0	0.126	0.281	9.677	1.535	1.57	1.475	3.405

The use of organic nitrogen rust inhibitors for boiler waters has become common practice. Two such inhibitors, octadecylamine acetate and cyclohexylamine, were subjected to distillation at the various pH values. No hydrolysis of the amino nitrogen was found at any of the pH values used. The initial 0.25 per cent of ammonia nitrogen found for octadecylamine acetate at pH 9.0, 11.0, and 14.0 is probably due to impurities in the compound. The most objectionable feature of these compounds is that they are distillable and produce a turbidity in the nesslerized sample. Even at the concentrations in which they are used in practice, a turbidity was obtained at the first 50-ml portions distilled.

REPORT OF COMMITTEE D-20

ON

PLASTICS*

Committee D-20 on Plastics, and its subcommittees, held three meetings during the year: at Atlantic City, N. J., on June 29 to July 1, 1955; at Cincinnati, Ohio, on November 14 to 16, 1955; and at Philadelphia, Pa., on February 15 to 17, 1956. The Advisory Committee also met at these times.

The committee participates in the work of Technical Committee 61 of the International Organization for Standardization (ISO), through its American Group for ISO/TC 61. Representatives from the United States attended the fifth meeting of ISO/TC 61 at Paris, France, July 8 to 13, 1955 and will attend the next meeting to be held at The Hague, The Netherlands, September 17 to 22, 1956. Notable progress was made by this committee in standardization for international trade. A report on the Paris meeting appeared in the October, 1955, ASTM BULLETIN, page 21.

Five Draft ISO Recommendations, dealing with the determinations of water absorption, temperature of deflection under load, acetone soluble matter in phenolic moldings, and apparent densities of molding powders, pourable and non-pourable, have been distributed for international balloting. Committee D-20 has ratified these five Draft ISO Recommendations and has transmitted the results of its letter ballot to the American Group for ISO/TC 61. C. H. Adams, of Monsanto Chemical Co., has been appointed chairman of the American Group, succeeding Robert Burns who has resigned.

The committee has reactivated Subcommittee IV on Optical Properties, with G. W. Ingle, chairman, because of renewed interest in optical measurements on plastics, especially films. The committee transferred the assignment of preparing test methods and specifications for reinforced plastic pipe from Subcommittee XVII on Plastic Pipe and Fittings to Subcommittee XVIII on Reinforced Plastics in the interest of homogeneity of assignments and personnel.

The committee adopted the following resolution:

> Whereas in Standards on plastics, the statement of test temperatures is preferably in degrees Centigrade, be it resolved that Committee D-20 regards with favor the rounding of the Centigrade-scale tolerance for the temperature of the Standard Laboratory Atmosphere to 1 C, and the changing of the parenthetical Fahrenheit equivalent to (1.8 F); and that this resolution be communicated to Committee E-1 on Methods of Testing.

The officers elected for the ensuing two-year term are as follows:

Chairman, Frank W. Reinhart.
First Vice-Chairman, A. C. Webber.
Second Vice-Chairman, A. G. H. Dietz.
General Secretary, J. B. DeCoste.
Membership Secretary, R. H. Carey.

RECOMMENDATIONS AFFECTING STANDARDS

The committee is presenting for publication 6 new tentatives and is recommending the revision of 6 tentatives, the tentative revision of 1 standard, the revision and reversion to tentative of 3 standards, the adoption as standards of 6 tentatives, the revision of 1 standard for immediate adoption, and the withdrawal of 2 tentatives. The standards affected, together with the revisions recommended, are given in detail in the Appendix.

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* **Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.**

The committee has considered all the other tentatives under its jurisdiction, not mentioned in the Appendix, and which have been published for two years or longer, and recommends that they be continued in their present status until further revisions in them are made as contemplated.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[1]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Mechanical Properties (R. M. Berg, chairman).—In progress are revisions, concerning speeds of testing for certain plastics, in the Tentative Methods of Test for Tensile Properties of Plastics (D 638 - 52 T) and for Flexural Properties of Plastics (D 790 - 49 T). Development of a tensile impact test has been undertaken. Work is progressing on a number of test methods applicable to films, including methods for modulus and for tear strength of thin films and methods for impact strength and for quantitative measurement of blocking and slip. The Rockwell-J hardness scale and the Durometer A, the Durometer D, and the Barcol hardness instruments are being standardized for nonrigid plastics.

Subcommittee III on Thermal Properties (C. H. Adams, chairman).—The intense activity on flammability tests during the year has resulted in significant revisions of the Standard Methods of Test for Flammability of Rigid Plastics over 0.050 in. in Thickness (D 635 - 44) and for Flammability of Plastics 0.050 in. and Under in Thickness (D 568 - 43) and in completion of a Tentative Method for Flammability of Flexible Thin Plastic Sheets. The revisions of these methods are included in this report. Methods for determining flammability of foams and ignition temperature of plastics are under study. Important liaison work on flammability is under way with a NEMA switch gear committee and with ASTM Committee E-5 on Fire Tests of Materials and Constructions. Limited interest in a stress-relaxation method led to dropping it. Work is in progress to broaden the applicability of the Zero-Strength-Time Test developed for polychlorotrifluoroethylene plastics. A task group has been formed to develop a method for flow of thermoplastic materials at high shear rates. Work on a parallel-plate plastometer method has been discontinued. A low-temperature brittleness method for film has been drafted for consideration by the Subcommittee. A softening point test based on the Vicat technique will be studied.

Subcommittee IV on Optical Properties (G. W. Ingle, chairman) is undertaking work on clarity of films and will cooperate with Subcommittee V on Permanence Properties in work on plastic color standards for calibration of laboratory aging test apparatus.

Subcommittee V on Permanence Properties (B. G. Achhammer, chairman) is developing a method employing fading of dyed plastic specimens for calibration of long-time exposures in carbon-arc laboratory aging equipment. Consideration is being given to development of a test method for laboratory aging of plastics by carbon-arc machines. The question of incorporating a long-time water absorption test in the Tentative Method of Test for Water Absorption of Plastics (D 570 – 54 T) is being evaluated. Revision of the Standard Method of Test for Colorfastness of Plastics to Light (D 620 – 49), and of the Standard Recommended Practice for Accelerated Weathering of Plastics Using the S-1 Bulb and Fog Chamber (D 795 – 49), in respect to specifying a shorter use time for the S-1 sun lamp has been initiated. Standardization of a laboratory aging method employing fluorescent sun lamps is being undertaken. The effect of angle of exposure of specimens in outdoor weathering tests is being studied. A new section on effects of nuclear radiation on plastics is being formed.

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[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee VII on Analytical Methods (H. E. Riley, chairman) is continuing its efforts to prepare adequate analytical methods for density of polyethylene; for free formaldehyde, ammonia and phenol in phenolic moldings; and for particle size in vinyl resins. Consideration is being given to revision of Standard Method of Test for Specific Viscosity of Vinyl Chloride Polymers (D 1243 – 54 T) in order to permit use of an alternate solvent and to permit expression of viscosity as inherent viscosity. Another active project is on the development of methods for determining the degree of dispersion and the content of carbon black in polyethylene.

Subcommittee VII on Research (A. G. H. Dietz, chairman) has continued to arrange for presentation of papers on subjects of current interest to the committee. There are no plans for publication of these papers as a group. Individual papers may be submitted for publication by ASTM or elsewhere.

At the fall meeting four papers were presented:

"A Study of Modulus *versus* Density Relationship for Polyethylene Resins," by R. Supnik and C. H. Adams of Monsanto Chemical Co.;

"Evaluation of Impact Properties of High Polymers by Tension Impact Tests," by C. G. Bragaw, Jr. of du Pont Polychemicals Department;

"The Effect of Notch Radius Variations on Izod Impact Testing," by G. B. Jackson and C. H. Adams of Monsanto Chemical Co.; and

"The Determination of Resin Content in Calcium Carbonate-Filled Glass Fiber Polyester Laminates," by S. D. Toner and Frank W. Reinhart of the National Bureau of Standards.

At the spring, 1956, meeting the following three papers were presented:

"Effects of Molding Pressure on the Strength Properties of Some Glass Fiber Reinforced Plastics," by S. D. Toner, Irvin Wolock and F. W. Reinhart of the National Bureau of Standards,

"A Film Toughness Tester," by R. D. Spangler of du Pont Polychemicals Department, and

"Comparison of Some Methods for Determining Young's Modulus in Flexure," by H. S. Loveless of American Cyanamid Co.

Subcommittee IX on Molds and Molding Processes (J. L. Williams, chairman) is revising the Tentative Recommended Practice for Injection Molding of Specimens of Thermoplastic Materials (D 1130 – 50 T). A mold is being developed for compression molding of sheet for test specimen preparation and a molding procedure for preparation of polyesters for testing is being prepared.

Subcommittee X on Definitions, Nomenclature, and Significance of Tests (P. E. Willard, chairman) is continuing to add to and to revise the Tentative Definitions of Terms Pertaining to Plastics (D 883 – 55 T) and Tentative Nomenclature of Descriptive Terms Pertaining to Plastics (D 675 – 55 T) so as to keep definitions and nomenclature current. Definitions are being developed for polyetheylene, polymethylene, and polypropylene, as well as a number of other terms.

Subcommittee XIV on Conditioning (G. H. Mains, chairman).—A tentative revision of the Standard Methods of

Conditioning Plastics and Electrical Insulating Materials for Testing (D 618 – 54) has been completed to provide three additional test temperatures, and to explain differences in nomenclature and reconcile differences in temperature tolerances between this method and the methods in MIL-P specifications and in other ASTM methods. The work of this subcommittee is a joint activity with Committee D-9 on Electrical Insulating Materials.

Subcommittee XV on Thermoplastic Materials (A. C. Webber, chairman).—Three new specifications are being developed: one for rigid vinyl chloride plastics, to include a normal and a high impact grade; one for styrene-rubber plastics; and one for polytetrafluoroethylene plastics. The subcommittee is revising the Tentative Specifications for Nylon Injection-Molding and Extrusion Compositions (D 789 – 53 T) to include the available commercial compositions. Considerable work is under way to prepare specifications and characterizing test methods applicable to the rapidly growing numbers of types of ethylene plastics. Work is in progress to determine the feasibility of developing tests for specifying a grade of plasticizer suitable for vinyl chloride plastic compounds for use in electrical applications. A new Section M is being formed as a consolidation of the work on specifications for all cellulose ester plastics and to prepare specifications on cellulose propionate plastics.

Subcommittee XVI on Thermosetting Materials (G. D. Johnston, chairman) is preparing specifications for a new glass-mat filled type of polyester resin and for new allyl plastic. Proposed revisions in Tentative Specifications for Phenolic Molding Compounds (D 700 – 55 T) are being considered, and work is in progress on specifications for potting compounds.

Subcommittee XVII on Plastic Pipe and Fittings (J. W. Sinner, chairman).—A subcommittee ballot on dimensions and tolerances for vinyl plastic pipe is in progress, and a proposed schedule for butyrate pipe has been voted on. A proposed polyethylene pipe standard is under study and a task group is studying the applicability of the Canadian pipe-cracking test specification. Schedules of Iron Pipe Size (IPA), and Solvent Welded Pipe, (SWP), dimensional standards are being prepared for styrene-rubber pipe. Development of specifications for the several classes of thermoplastic-pipe fittings is in progress. A task group on burst testing has been organized and the sections on burst and creep methods have been amalgamated into Section G on Hydrostatic Pressure Testing. Section E on Reinforced Plastic Pipe and its work has been transferred to Subcommittee XVIII on Reinforced Plastics in the interests of homogeneity of projects and personnel. The work of this subcommittee is a joint effort with the Society of the Plastics Industry.

Subcommittee XVIII on Reinforced Plastics (R. B. Crepps, chairman) has prepared a number of test methods and specifications for subcommittee ballot. These include specifications for glass fabric reinforced epoxy resin laminates; dimensional standards for reinforced plastic pipe; methods for effects of soil water on pipe and evaluation of fittings; methods for determining light transmission of corrugated panels; acid number of polyester resins and epoxy content of epoxy resins; and a loading test for reinforced plastic panels. Other projects in progress concern methods for viscosity of polyester and epoxy resins, curing characteristics of polyester resins, and specification requirements for glass fabrics, rovings and chopped strand used as reinforcements.

Subcommittee XIX on Plastic Film and Sheeting (Bill Williams, chairman) has drafted a specification for nonrigid vinyl chloride plastic film for subcommittee ballot. Task groups are preparing specifications for extruded acrylic sheet and for rigid vinyl chloride film. The subcommittee is developing specifications, for general purpose, electrical, and outdoor barrier types of polyethylene film and for styrene and styrene-acrylonitrile plastic sheet. Round-robin tests to determine ranges of property values needed for a styrene-rubber plastic sheeting specification have been initiated. The subcommittee is revising the Standard Specifications for Cellulose Acetate Plastic Sheet (D 786 - 49) with respect to specifying optical and surface characteristics with cooperation from Subcommittee IV on Optical Properties.

Subcommittee XX on Cellular Plastics (C. H. Alexander, chairman) is being organized, in stand-by status, pending specific assignments.

This report has been submitted to letter ballot of the committee, which consists of 213 members; 131 members returned their ballots, of whom 120 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

R. K. Witt,
Chairman.

G. M. Armstrong,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-20 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Specifications for:

Tetrafluoroethylene Resin Molding and Extrusion Materials (D 1457 - 56 T).

Revision of Tentative Method of Test for:

Tensile Properties of Plastics (D 638 - 52 T), and
Tensile Properties of Thin Plastic Sheets and Films (D 882 - 54 T).

These recommendations were accepted by the Standards Committee on December 17, 1956, and the new specifications and revised methods appear in the February, 1956 Compilation of ASTM Standards on Plastics.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS ON PLASTICS

In this Appendix are listed six new tentatives and proposed revisions in certain standards and tentatives which are referred to earlier in this report. These standards and tentatives appear in their present form in the 1955 Book of ASTM Standards, Part 6.

NEW TENTATIVES

Committee D-20 recommends that the following six new tentatives be accepted for publication as tentative as appended hereto:[1]

Tentative Specifications for Nonrigid Polyvinyl Chloride and Copolymer Molding and Extrusion Compounds:

These specifications are the result of revisions, in accordance with present commercial compounds, and combination into a single specification of the two tentative specifications for Nonrigid Vinyl Chloride-Acetate Resin Plastics (D 742 – 46 T) and Nonrigid Polyvinyl Chloride Plastics (D 744 – 49 T).

Tentative Specifications for Polychlorotrifluoroethylene Molding and Extrusion Materials:

These specifications cover four commercial grades of polychlorotrifluoroethylene plastics, and includes three new methods of tests developed specifically for identification of these grades. It is the intent of the committee that these methods be revised for more general applicability and that they be eventually separated from this specification.

Tentative Specifications for Styrene Acrylonitrile Copolymer Molding and Extrusion Materials:

These specifications characterize a general purpose type of commercial material. Specifications of other types will be added as needed.

Tentative Method of Test for Flammability of Flexible Thin Plastic Sheeting:

This method fills a need for a flammability test suitable for comparing relative burning rates of thin plastic sheeting for control testing or for relative flammability rating. It is not in its present form suitable for specifications testing.

Tentative Method of Test for Gas Transmission Rate of Plastic Sheeting:

This method provides an apparatus and an operating technique for determining the rates of transmission of gases through plastic sheeting, a property of great importance in the packaging industry, especially the packaging of foodstuffs.

Tentative Recommended Practice for Outdoor Weathering of Plastics:

This practice provides a needed procedure for exposure of plastics to weather with details relating to methods of exposure, general procedures recommended, and kinds of climatological information to be recorded.

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[1] The new and revised tentatives appear in the 1956 Supplement to Book of ASTM Standards, Part 6.

REVISION OF TENTATIVES

The committee recommends that the following six tentatives be revised as indicated and continued as tentative:

Tentative Specifications for Polystyrene Molding Compounds (D 703 - 49 T):

This revision, appended hereto as complete revised specifications,[1] eliminates the requirements of solution viscosity and methanol soluble content, which are no longer needed; brings certain properties requirements into line with experience on present types; and provides for including a fourth type of molding or extrusion material.

Tentative Specifications for Melamine-Formaldehyde Molding Compounds (D 704 - 55 T):

Revise to include an additional Type 9 as follows:

Section 2.—Reword first sentence and add Type 9 as follows:

2. These specifications cover the following nine types of melamine-formaldehyde molding compounds:

TYPE 9.—A general purpose material with alpha-cellulose and mineral filler, suitable for general mechanical and electrical applications.

TABLE I.—DETAIL REQUIREMENTS FOR COMPRESSION MOLDED TEST SPECIMENS.

(Revision of Table I, D 704 - 55 T)

		Type 9
Specific Gravity 23/23 C (73.4/73.4 F)	min	1.46
	max	1.50
Flexural Strength, min, psi		7500
Flexural Deflection, min, in		0.06
Impact Strengh (Izod), min, ft lb per in. of notch		0.28
Water Absorption (24 hr, immersion), max weight gain, per cent		0.55
Mold Shrinkage, max, in. per in		0.007
Dielectric Strength min, v per mil	Short-time test	350
	Step-by-step test	250
Loss Factor, max	60 cycles per sec	0.55
	1,000 cycles per sec	0.25
	1,000,000 cycles per sec	0.20
Arc Resistance, min, sec		115

Table I.—Add values for Type 9 as shown in the accompanying Table I.

Tentative Specifications for Methacrylate Molding and Extrusion Compounds (D 788 - 48 T):

Revise as appended hereto.[1] Changes comprise revisions in Sections 2, 3, 4, and 6 and in Table I.

Tentative Method of Test for Resistance of Plastics to Chemical Reagents (D 543 - 52 T):

Section 4.—Add a new Paragraph (*o*) to read as follows:

(*o*) *Distilled Water.*—Freshly prepared.

Tentative Nomenclature of Descriptive Terms Pertaining to Plastics (D 675 - 55 T):

Revise descriptions for the following terms:

Mold Seam.—Line on a molded or laminated piece, differing in color or appearance from the general surface, caused by the parting line of the mold.

Gloss.—Delete the term and the definition.

Tentative Definitions of Terms Relating to Plastics (D 883 - 55 T):

Add the following new definitions:

Cold Molding.—The shaping of compounds in a mold under pressure followed by transfer of the part to a heating medium for curing.

Degradation, n.—A deleterious change in the chemical structure of a plastic. (See also Deterioration.)

Extrusion, n.—A method whereby heated or unheated plastic forced through a shaping orifice becomes one continuously formed piece.

Gel, n.—(*1*) A semisolid system consisting of a network of solid aggregates in which liquid is held.

(*2*) The initial jelly-like solid phase that develops during the formation of a resin from a liquid.

NOTE.—Both types of gel have very low strengths and do not flow like a liquid. They are soft, flexible and will rupture under their own weight unless supported externally.

Gel Point.—The stage at which a liquid system becomes a gel. (See second definition of Gel.)

NOTE.—This may be caused by such factors as continuing polymerization, change in temperature, or loss of solvent.

Polyvinyl Acetate.—A resin prepared by the polymerization of vinyl acetate alone.

Polyvinyl Alcohol.—Polymers prepared by the hydrolysis of polyvinyl esters.

Polyvinyl Chloride.—A resin prepared by the polymerization of vinyl chloride alone.

Stress-crack, n.—External or internal cracks in a plastic caused by tensile stresses less than that of its short-time mechanical strength.

NOTE.—The development of such cracks is frequently accelerated by the environment to which the plastic is exposed. The stresses which cause cracking may be present internally or externally or may be combinations of these stresses. The appearance of a network of fine cracks is called crazing.

Syneresis.—The contraction of a gel.

NOTE.—This is usually evidenced by the separation of a liquid. (See first definition of Gel.)

Vinyl Acetate Plastics.—Plastics based on resins made by the polymerization of vinyl acetate or copolymerization of vinyl acetate with minor amounts (not over 50 per cent) of other unsaturated compounds.

Vinyl Alcohol Plastics.—Plastics based on resins made by the hydrolysis of polyvinyl esters or copolymers of vinyl esters.

Vinyl Chloride Plastics.—Plastics based on resins made by the polymerization of vinyl chloride or copolymerization of vinyl chloride with minor amounts (not over 50 per cent) of other unsaturated compounds.

TENTATIVE REVISION OF STANDARD

Committee D-20, jointly with Committee D-9 on Electrical Insulating Materials, recommends a tentative revision of the Standard Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (D 618 – 54), as set forth in the D-9 Report. This tentative revision consists of the addition of three new standard test temperatures, and revisions to reconcile differences in temperature tolerances and in nomenclature between this and other conditioning standards.

REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommends that the following three standards be revised as indicated and reverted to tentative status:

Standard Specifications for Cast Methacrylate Plastic Sheets, Rods, Tubes, and Shapes (D 702 – 46): Revise as appended hereto.[1] Revisions comprise extensive changes in Sections 2, 3, 4, and 6. Revisions in Section 4 include changes in Table II and in Fig. 2.

Standard Method of Test for Flammability of Plastics 0.050 in. and Under in Thickness (D 568 – 43): Revise for improved reproducibility as appended hereto.[1]

Standard Method of Test for Flammability of Plastics 0.050 in. and Over in Thickness (D 635 – 44): Revise for improved reproducibility as appended hereto.[1]

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following six tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Specifications for:

Enclosures and Servicing Units for Tests Above and Below Room Temperature (**D 1197 – 54 T**), (Jointly with Committee D-9 on Electrical Insulating Materials.)

Tentative Methods of Test for:

Impact Resistance of Plastics and Electrical Insulating Materials (**D 256 – 54 T**),

Resistance of Transparent Plastics to Surface Abrasion (**D 1044 – 54 T**),

Warpage of Sheet Plastics (**D 1181 – 51 T**),

Resistance to Abrasion of Plastics Materials (**D 1242 – 52 T**), and

Tentative Recommended Practice for:

Long-Time Creep or Stress-Relaxation of Plastics under Tension or Compression Loads at Different Temperatures (**D 674 - 51 T**).

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption the following revision of the Standard Methods of Test for Resistance of Plastics to Accelerated Service Conditions (**D 756 - 50**) in conformity with the policy of referring test temperatures to the Centrigrade scale, and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that this revision may be referred to letter ballot of the Society.

Section 3.—In Paragraph (*b*), change "plus or minus 2 F" to read "plus or minus 1 C (1.8 F)." In Paragraph (*g*), change "plus or minus 5 F" to read "plus or minus 3 C (5.4 F)."

Section 8.—In Paragraph (*a*), lines 3 and 5, change "24 hr at 140 F" to read "24 hr at 60 C (140 F)." In Paragraph (*c*), line 3, and Paragraph (*f*), last line, change "at 140 ± 2 F" to read "at 60 ± 1 C (140 ± 1.8 F)."

Section 9.—In Paragraph (*a*), line 3, change "72 hr at 140 F" to read "72 hr at 60 C (140 F)." In Paragraph (*c*), line 2, change "140 ± 2 F" to read "60 ± 1 C (140 ± 1.8 F)."

Section 10.—In Paragraph (*a*), lines 3 and 5, change "160 F" to read "70 C (158 F)." In Paragraph (*c*), line 3, and Paragraph (*f*), line 4, change "160 ± 2 F" to read "70 ± 1 C (158 ± 1.8 F)."

Section 11.—In Paragraph (*a*), lines 3 and 4, change "175 F" to read "80 C (176 F)." In Paragraph (*c*), line 3, and Paragraph (*f*), line 4, change "175 ± 2 F" to read "80 ± 1 C (176 ± 1.8 F)."

Section 12.—In Paragraph (*a*), lines 3 and 6, change "175 F" to read "80 C (176 F);" in lines 5 and 7, change "24 hr at −40 F or −70 F as specified" to read "24 hr at −40 C (−40 F) or −57 C (−70.6 F) as specified." In Paragraph (*c*), line 3, change "175 ± 2 F" to read "80 ± 1 C (176 ± 1.8 F)." In Paragraph (*e*), line 4, change "−40 ± 4 F" to read "−40 ± 2 C (−40 ± 3.6 F);" in line 5, change "−70 ± 4 F" to read "−57 ± 2 C (−70.6 ± 3.6 F)." In Paragraph (*h*), line 4, change "175 ± 2 F" to read "80 ± 1 C (176 ± 1.8 F)." In Paragraph (*j*), line 4, change "−40 ± 4 F or −70 ± 4 F as specified" to read: "−40 ± 2 C (−40 ± 3.6 F), or −57 ± 2 C (−70.6 ± 3.6 F)."

Section 13.—In Paragraph (*a*), line 3, change "100 F" to read "38 C (100.4 F);" in line 5, change "140 F" to read "60 C (140 F)." In Paragraph (*c*), line 2, change "100 ± 2 F" to read "38 ± 1 C (100.4 ± 1.8 F)." In Paragraph (*f*), line 4, change "140 F" to read "60 C (140 F)."

Section 14.—In Paragraph (*a*), lines 3 and 5, change "120 F" to read "49 C (120.2 F)." In Paragraph (*c*), line 2, change "120 ± 2 F" to read "49 ± 1 C (120.2 ± 1.8 F)." In Paragraph (*f*), line 5, change "120 F" to read "49 C (120.2 F)."

WITHDRAWAL OF TENTATIVES

The committee recommends the withdrawal of the following two tentatives:

Tentative Specifications for:

Nonrigid Vinyl Chloride-Acetate Resin Plastics (**D 742 - 46 T**),

Nonrigid Vinyl Chloride Plastics (**D 744 - 49 T**).

These specifications are superseded by the new Tentative Specifications for Nonrigid Polyvinyl Chloride Molding and Extrusion Compounds, referred to earlier in the report.

REPORT OF COMMITTEE D-21
ON
WAX POLISHES AND RELATED MATERIALS*

Committee D-21 on Wax Polishes and Related Materials held one meeting during the year: at Buffalo, N. Y., on February 27 and 28, 1956.

Additions and changes during the year brought the total membership of the committee to 70, of whom 33 are classified as producers, 14 as consumers, and 23 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, W. W. Walton.
Vice-Chairman, J. V. Steinle.
Secretary, B. S. Johnson.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1955 Annual Meeting, Committee D-21 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Method of Test for:

Acid Number (Empirical) of Natural Waxes (D 1386 - 55 T), and
Saponification Number (Empirical) of Natural Waxes (D 1387 - 55 T).

These recommendations were accepted by the Standards Committee on July 25, 1955, and the new methods appear in the 1955 Book of ASTM Standards, Part 4.

NEW TENTATIVES

The committee recommends that the following methods be accepted for publication as tentative as appended hereto:[1]

Method of Test for:

Flash Point of Solvent-Type Liquid Waxes,

Methods of Application of:

Emulsion Floor Polishes to Substrates for Testing Purposes.

REVISIONS OF TENTATIVE

The committee recommends that the Tentative Method of Test for Paraffin-Type Hydrocarbons in Carnauba Wax (**D 1342 - 54 T**)[2] be revised as follows and continued as tentative:

Section 4.—In Paragraph (*a*), line 10, change "hot (75 to 80 C) heptane" to read "heptane at its boiling point."

In Paragraph (*b*), the first sentence, change "700 ml of warm (50 to 55 C) heptane" to read "700 ml of boiling heptane." In the second sentence, place a period after the word "alumina" and delete the phrase "and adjust the stopcock so that the percolate collects in a beaker placed under the funnel at a rate not exceeding 600 ml per hr." Add the following as the third sentence: "Collect the percolate in a beaker placed under the funnel with the stopcock in a position to permit maximum flow through the column." In the present third sentence, change "50 to 55 C" to read "at approximately the boiling point of heptane." Following this sentence insert the following sentence: "The temperature of the percolate issuing from the funnel stem should be at 50 to 55 C."

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] The new tentatives appear in the 1956 Supplement to Book of ASTM Standards, Part 4.

[2] 1955 Book of ASTM Standards, Part 4.

In Paragraph (*c*), the first sentence, change "additional warm (50 to 55 C) heptane" to read "additional heptane at within 5 C of its boiling point."

In Paragraph (*e*), the last sentence, delete "with the flow rate through the column reduced to 400 ml per hr."

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following methods be continued as tentative without revision:

Tentative Method of Test for:

Total Ash and Silica in Water-Emulsion Waxes **(D 1288 - 53 T)**, and

Sediment in Water-Emulsion Waxes by Centrifuge **(D 1290 - 53 T)**.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature (J. T. Hohnstine, chairman) is preparing a glossary of terms relating to wax polishes.

Subcommittee II on Raw Materials (Melvin Fuld, chairman) prepared the revision of the Tentative Method of Test D 1342 - 54 T set forth previously in this report.

The subcommittee is working on revisions of Tentative Methods D 1386 - 55 T and D 1387 - 55 T for the acid number and saponification number of natural waxes. In addition, several task groups are actively working on the use of the Pensky-Martens flash tester as a method of determining adulteration of Carnauba wax, the determination of sulfur in Carnauba wax, as well as several other test methods.

Subcommittee III on Physical and Chemical Testing (Gerald DeNapoli, chairman) has developed the Method for Determining the Flash Point of Solvent-Type Liquid Waxes which is referred to earlier in this report for publication as tentative.

The subcommittee is currently developing a method for the determination of non-volatile matter in solvent waxes and a task group is working on a test method for stability of water emulsion polishes.

Subcommittee IV on Performance Tests (C. S. Kimball, chairman).—The section on slip resistance is continuing its development of a suitable secondary standard for the calibration of both the Sigler and James slip testers.

The section on service life is developing a Method of Test for Water Spotting of Emulsion Floor Polishes and also is working on a method for determining the removability properties of floor waxes.

The section on appearance has developed the Methods of Application for Water Emulsion Waxes to Substrates referred to earlier in the report for publication as tentative. The section is preparing a recommended practice for the determination of gloss of floor waxes.

Subcommittee V on Specifications (William Joy, chairman) is preparing specifications for industrial type water emulsion floor waxes. Completion of this work awaits the development by the other subcommittees of test methods for several of the properties involved.

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

This report has been submitted to letter ballot of the committee, which consists of 70 members; 39 members have returned their ballots, all of whom voted affirmatively.

Respectfully submitted on behalf of the committee,

W. W. WALTON,
Chairman.

BAYARD S. JOHNSON,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-21 presented to the Society through the Administrative Committee on Standards the recommendation that the proposed Method of Test for 60-deg Specular Gloss of Emulsion Floor Polish be accepted for publication as tentative. This recommendation was accepted by the Standards Committee on September 5, 1956, and the new tentative method appears in the 1956 Supplement to Book of ASTM Standards, Part 4, bearing the designation D 1455 – 56 T.

REPORT OF COMMITTEE D-22

ON

METHODS OF ATMOSPHERIC SAMPLING AND ANALYSIS*

Committee D-22 on Methods of Atmospheric Sampling and Analysis held two meetings during the year: on September 12, 1955, in Minneapolis, Minn., and on February 27 and 28, 1956, in Buffalo, N. Y.

The membership of the committee consists of 62 members, of whom three are consulting members.

The officers elected for the ensuing term of two years are as follows:

Chairman, L. C. McCabe.
Vice-Chairman, Morris Katz.
Secretary, W. A. Cook.

Recommendations Accepted by the Administrative Committee on Standards

Subsequent to the 1955 Annual Meeting, the committee presented to the Society through the Administrative Committee on Standards the Tentative Method of Test for Odor Concentration in Air and Gases (Dilution Method) (D 1391 - 56 T). This recommendation was accepted by the Standards Committee on April 2, 1956.

Activities of Subcommittees

Subcommittee I on Nomenclature and Units (F. A. Patty, chairman) is working on definitions for a number of terms to be recommended for addition to the Tentative Definition of Terms Relating to Atmospheric Sampling and Analysis (D 1356 - 55 T). Another active project is the development of a list of units and conversion factors used in air analysis.

Subcommittee II on Methods of Sampling (Leslie Silverman, chairman) has completed a draft of a method of sampling for gases and vapors which is currently under consideration. A method of sampling for particulate matter has been drafted. Included are requirements for dust-fall jars and for filter media. Stack sampling is another active project.

Subcommittee III on Analytical Methods (J. Cholak, chairman) prepared the Tentative Method of Test for Odor Concentration in Air and Gases (D 1391 - 56 T) referred to above. Active projects are in progress on methods of analysis for oxides of nitrogen, methods for determining fluorides in the atmosphere, methods for total sulfur as sulfate, methods of analysis for stack gases and particulate matter, and methods for determining ozone.

Subcommittee IV on Instrumentation (M. D. Thomas, chairman) has drafted a method of test for mass concentration of particulate matter in the atmosphere (light scattering method).

Another method for particulate matter based on optical density of filtered deposit is being drafted. An alternate method for continuous analysis and automatic recording of sulfur dioxide in the atmosphere referred to as the electrolysis method is being considered as an addition to Tentative Method D 1355.

This report has been submitted to letter ballot of the committee, which consists of 62 members; 43 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

L. C. McCabe,
Chairman.

H. H. Schrenk,
Secretary.

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* **Presented at the Fifty-ninth Annual Meeting of the Society, June 18–22, 1956.**

REPORT OF COMMITTEE D-23

ON

CELLULOSE AND CELLULOSE DERIVATIVES*

Committee D-23 on Cellulose and Cellulose Derivatives held one meeting during the year: in Minneapolis, Minn., on September 12, 1955. The Executive Subcommittee met also in New York City on February 22, 1956.

The committee consists of 46 members of whom 14 are classified as producers, 11 as consumers, and 21 as general interest members.

The officers elected for the ensuing two-year term are as follows:

Chairman, F. A. Simmonds.
Vice-Chairman, O. A. Battista.
Secretary, W. W. Becker.

NEW TENTATIVES

The committee recommends that the following two methods of test be accepted for publication as tentative as appended hereto.[1]

Tentative Method of Test for:

Pentosans in Cellulose, and
Sodium Carboxymethylcellulose.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following three tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Method of:

Testing Cellulose Acetate (**D 871 - 54 T**),[2]
Testing Methylcellulose (**D 1347 - 54 T**),[2] and
Test for Moisture in Cellulose (**D 1348 - 54 T**).

ADOPTION OF TENTATIVES AS STANDARDS WITH REVISION

The committee recommends that the following two tentatives be approved for reference to letter ballot of the Society for adoption as standard, with the following editorial changes and corrections:

Specifications and Methods of Test for Soluble Cellulose Nitrate (D 301 - 54 T):[2]

Section 4 (*d*).—In the last line, change "(*b*)" to "(*c*)."

Section 5.—In the second line, change "4(*a*)" to "4(*b*)."

Section 4 (*c*).—Add the following note:

NOTE.—Information on the availability of a suitable fusible plug assembly may be obtained from ASTM Headquarters.

Viscosity of Cellulose Derivatives by Ball-Drop Method (D 1343 - 54 T)[2]

Section 4 (*a*).—Replace the first sentence with the following:

Dry the sample and prepare a solution as specified for the particular material. Such instructions are given in Section 11 of Specifications and Methods of Test for Soluble Cellulose Nitrate (ASTM Designation: D 301)[5], in Section 12 of the Methods of Testing Cellulose Acetate (ASTM Designation: D 871)[5], and in Section 32 of Methods of Testing Cellulose Acetate Butyrate (ASTM Designation: D 817).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17-22, 1956.

[1] The new tentatives appear in the 1956 Supplement to Book of ASTM Standards, Part 4.

[2] 1955 Book of ASTM Standards, Part 4.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature and Specifications (Kyle Ward, Jr., chairman) has prepared definitions for 70 terms used in the field of cellulose and cellulose derivatives and they are being circulated to Committee D-23 for consideration.

Subcommittee II on Cellulose (O. A. Battista, chairman) has recommended that the Tentative Methods of Test for Moisture in Cellulose (D 1348 – 54 T) be adopted as standard.

This subcommittee prepared the proposed tentative method of test for pentosans in cellulose, which is recommended for publication as tentative as mentioned earlier in this report.

Interlaboratory collaborative testing of proposed procedures have been organized by Sections on Absorption and Color of Chemical Cellulose, on Ash Constituents in Cellulose, and on Extractives in Cellulose.

Progress is being made on improved tests for cold alkali solubility and disperse viscosity of cellulose. A Karl Fischer test for moisture in cellulose is under consideration.

New sections on methods of test for functional groups and on chromatographic techniques for cellulose have been organized.

Subcommittee III on Organic Esters (L. B. Genung, chairman). The subcommittee is studying the carbanilation method for the direct measurement of hydroxyl content in cellulose derivatives.

Subcommittee IV on Inorganic Esters (C. B. Gilbert, chairman) has recommended that the Tentative Specifications and Methods of Test for Soluble Cellulose Nitrate (D 301 – 54 T) be adopted as standard.

Subcommittee V on Cellulose Ethers (R. W. Swinehart, chairman) has recommended that the Tentative Methods of Testing Methylcellulose (D 1347 – 54 T) be adopted as standard.

This subcommittee prepared the proposed tentative method of testing sodium carboxymethylcellulose which relates to moisture and viscosity and is recommended for publication as tentative as mentioned earlier in this report.

The work on an assay procedure for determining the purity of sodium carboxymethylcellulose is continuing.

A task group on hydroxyalkyl cellulose has been formed with R. A. Leonard as chairman.

Subcommittee VI on Statistics (E. J. Delate, chairman) sponsored a round table discussion on the statistical aspects of an interlaboratory study on determination of acetyl in cellulose acetate at the September meeting. It was brought out that a clear statement of the objectives of an interlaboratory test is important and the design of the check should be such that the questions asked can be answered with a high degree of confidence with minimum testing effort.

Work on the compilation of a manual on statistics is in progress. An interlaboratory test among five laboratories for evaluating two proposed assay methods for sodium carboxymethylcellulose has been designed.

This report has been submitted to letter ballot of the committee, which consists of 46 members; 31 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

F. A. SIMMONDS,
Chairman.

W. W. BECKER
Secretary.

REPORT OF COMMITTEE E-1

ON

METHODS OF TESTING*

Committee E-1 on Methods of Testing held a meeting at Society headquarters in Philadelphia, Pa., on May 3, 1956.

Meetings were held of 13 E-1 subcommittees and task groups during ASTM Committee Week in Buffalo, N. Y., on February 27, 28, and 29, 1956.

Subcommittee 17 on Thermometers held two meetings: in Washington, D. C., on October 7, 1955, and in Rochester, N. Y., on May 7 and 8, 1956.

Subcommittee 21 on Metalware Laboratory Apparatus held a meeting on December 6, 1955, in Philadelphia, Pa.

A new Subcommittee on Microchemical Apparatus under the chairmanship of A. Steyermark was organized during the year. Its personnel includes that of the Committee on Microchemical Apparatus of the Division of Analytical Chemistry of the American Chemical Society. Consideration will be given to the entire field of microchemical apparatus including glassware, metalware, filters, etc. The design of microchemical apparatus is, in general, closely related to and determined by its manner of use. Later, it may be found desirable to undertake the development of microchemical methods.

On the recommendations of Subcommittees 17, 18, and 19, which are concerned with laboratory apparatus, Committee E-1 is recommending active participation by the United States in the work of Technical Committee 48 on Laboratory Glassware and Related Apparatus of the International Standards Organization rather than as an "Observer Member."

The officers elected for the ensuing term of two years are as follows:

Chairman, J. R. Townsend.

Vice-Chairman, A. C. Webber.

Committee E-1 is sponsoring two Symposiums on the following subjects at the 1956 Annual Meeting:

Symposium on Tension Testing of Non-Metallic Materials,[1] sponsored by Subcommittee 4 on Tension Testing, comprising 6 papers to be presented on June 20.

Symposium on pH Measurement,[2] sponsored by Subcommittee 22 on Hydrogen Ion Determinations comprising 7 papers to be presented in two sessions on June 19.

At the 1956 Annual Meeting, there will also be a Papers Session on Various Complex Rheological Problems on June 22. This session is being held under the joint auspices of the Society of Rheology and the ASTM.

Plans are under way for a Symposium on Particle Size Measurement to be held at the 1958 Annual Meeting under the auspices of Subcommittee 11 on Sub-Sieve Testing. L. T. Work is chairman of the Symposium Committee.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1955 Annual Meet-

*** Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.**

[1] Issued as separate publication, *ASTM STP No. 194.*

[2] Issued as separate publication, *ASTM STP No. 190.*

ing, Committee E-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specification for:

ASTM Hydrometers (E 100 - 54 T).

Revision of Tentative Recommended Practice for:

Characteristics of Standard Carbon Arc Accelerated Weathering Unit (E 42 - 42 T), with a change in title to read "Tentative Recommended Practice for Operation of Light and Water Exposure Apparatus (Carbon-Arc Type) for Artificial Weathering Tests."

Tentative Revision of Standard Specification for:

ASTM Thermometers (E 1 - 55).

The revision of E 42 was accepted by the Standards Committee on December 19, 1955, and it appears in Parts 4, 6 and 7 of the 1955 Book of ASTM Standards. The remaining recommendations were accepted on September 15, 1955; the revision of E 100 appears in Parts 3 and 5 of the 1955 Book of Standards, and the tentative revision of E 1 appears in Part 5.

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends that the Standard Specifications for ASTM Thermometers (**E 1 - 55**) be revised as follows, and accordingly asks for a favorable nine-tenths vote at the Annual Meeting in order that this recommendation may be submitted to letter ballot of the Society:

Section 5.—Add the following as Paragraph (*b*), and designate the present section as Paragraph (*a*):

(*b*) In the case of the high-temperature thermometers, the bulb of the thermometer may be heated as now specified in Table I, provided that the temperatures of use do not exceed 760 F and that the thermometers are not continuously exposed to temperatures above 700 F.

Table I.—In the ASTM Stormer Viscosity Thermometer 49 C change the specified standardization temperatures from "At average temperature 25 C of emergent mercury column" to read "At 25 C and 50 C."

Following the June meeting, the committee plans to recommend a number of tentative thermometer specifications for inclusion in Standard E-1. These will include a set of solidification point thermometers, a vegetable oil flash point thermometer, and two tank gaging thermometers.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the tentative methods under its jurisdiction which have been published for two or more years without revision be continued as tentative. Action on revisions in several of these tentatives will be taken at the June meeting as mentioned later under the summary of subcommittee activities. These revisions will be submitted to the Society later in the year through the Administrative Committee on Standards.

The recommendation in this report has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee 1 on Calibration of Mechanical Testing Machines and Apparatus (B. L. Wilson, chairman).—Subcommittee 1 is completing revisions in the Tentative Methods of Verification of Testing Machines (E 4 - 55 T), and Verification of Calibration Devices for Verifying Testing Machines (E 74 - 55 T). The Tentative Method of Verification and Classification of Extensometers (E 83 - 52 T) is being continued as tentative.

Subcommittee 2 on Effect of Speed in

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[3] The letter ballot vote on this recommendation was favorable; the results of the vote are on record at ASTM Headquarters.

Mechanical Testing (D. E. Parsons, chairman).—Mr. B. L. Wilson resigned as secretary of Subcommittee 2 and chairman of the Task Group on Bibliography and Mr. L. K. Irwin was appointed to these offices.

The Task Group on Bibliography has started a second supplement to bring the bibliography up to date. This material may be ready for publication in 1956.

The Task Group on Metallic Materials (F. M. Howell, chairman) is considering the effects of speed on stainless steel and is preparing a report covering some recent investigations.

The Symposium on Speed of Testing of Non-Metallic Materials held at the 1955 Annual Meeting will shortly be issued as *Special Technical Publication 185*.

A task group was organized with H. L. Fry as chairman to explore the possibility of defining terms such as "impact", "high rate of straining", "rapid loading", etc. for use in specifications and technical reports.

Subcommittee 3 on Elastic Strength of Materials.—G. R. Gohn was appointed as chairman to replace F. M. Howell. Subcommittee 3 is developing a method of test for the determination of Poisson's ratio for sheet material at room temperature. This method is being prepared by a task group under the chairmanship of W. Ramberg. A second task group under the chairmanship of W. N. Findley is reviewing the various definitions of terms applying to the mechanical testing of non-metallic materials with a view towards their incorporation in Definitions E-6.

Subcommittee 4 on Tension Testing.—F. M. Howell was appointed to replace R. L. Templin as chairman.

A task group has been reviewing and studying differences in the tension testing procedures in Methods E 8 and A 370 and the forthcoming revisions of Federal Specification QQ-M-151a, together with the definitions in E-6. It has prepared rather extensive revisions of Method E 8 that are intended to eliminate conflicts among the various standards and specifications.

The need for sub-size specimens, particularly for testing radiated samples is being investigated.

Subcommittee 6 on Indentation Hardness (R. H. Heyer, chairman).—A task group is undertaking further revision of the Rockwell Hardness Methods E 18, including the addition of recommended minimum thicknesses of test specimens, and corrections for tests on rounds using the $\frac{1}{16}$-in. ball penetrator.

A task group is being organized, in cooperation with A-1 and representatives of the American Society for Metals, Society of Automotive Engineers, the Armed Services, and aircraft manufacturers, to reinvestigate hardness - tensile strength relationships for steel, especially in the high-hardness range.

A task group is undertaking the combination of all the hardness conversions into a single designation, maintaining separate tables for each class of materials as at present.

Subcommittee 7 on Impact Testing (W. W. Werring, chairman).—Subcommittee 7 is voting on a revised draft of the Tentative Methods of Impact Testing of Metallic Materials (E 23 - 47 T). This is draft No. 7 and there has been extensive detailed discussion on each draft. Considerable modernization of the method has been achieved together with additional detailed information on tolerances and accuracy. It is hoped that this can be finally approved at the June meeting.

The Symposium on Impact Testing held at the 1955 Annual Meeting has now been issued as *Special Technical Publication No. 176*.

Subcommittee 8 on Flexure Testing (H. F. Clemmer, chairman).—The proposal to refer the latest draft of the "Method for Making Three-Edge Bearing Test of Pipe" to a joint committee, composed of representatives of ASTM Committees A-3 on Cast Iron, C-4 on Clay Pipe, C-13 on Concrete Pipe, and C-15 on Manufactured Masonry Units, was approved by Committee E-1. These four committees have now been asked to appoint their representatives, and a meeting will be held in June. This committee is to be charged with reviewing the current draft and preparing a single standard method to be used in place of the various methods now included in the individual pipe specifications.

Subcommittee 14 on Conditioning and Weathering (A. C. Webber, chairman).—Methods E 42 were completely revised during the year and are now published as Tentative Recommended Practice for Operation of Light and Water-Exposure Apparatus (Carbon-Arc Type) for Artificial Weathering in Parts 4, 6 and 7 of the 1955 Book of ASTM Standards.

These basic procedures may now be used by the various technical committees as a reference in at least six standards for specific materials. Several are already in the process of revision, and at least one new method is being written. It is believed the present recommended practice will eliminate unnecessary repetition in the various printings, and improve correlation and communication in this important area of testing.

Subcommittee 15 on Correlation of Chemical Analyses (J. W. Stillman, chairman).—The most important matter under consideration by Subcommittee 15 during the year has been the Proposed Recommendations on the Form of ASTM Methods for Chemical Analysis which have been approved by letter ballot and were presented to Committee E-1 at the May meeting.

Subcommittee 15 has been trying to develop an opinion as to whether the ASTM should issue a combined publication of all analytical methods other than the Methods of Chemical Analysis for Metals. The first vote on this question was indecisive, and it was felt that no recommendation could be made at this time.

Subcommittee 17 on Thermometers (W. M. Wilhelm, chairman).—This subcommittee submitted the revisions in the Thermometer Specifications E-1 covered earlier in this report. As noted, additional changes in these specifications will be presented to the Administrative Committee on Standards later in the year. At that time, revisions will also be presented in the Tentative Method of Testing and Standardization of Etched-Stem Liquid-in-Glass Thermometers (E 77 - 52 T).

Subcommittee 18 on Hydrometers (R. D. Thompson, chairman).—Cooperative work has been carried on with ASTM Committee D-2 on Petroleum Products and Lubricants and with the American Petroleum Institute (API) in an effort to effect standardization of hydrometer specifications between these two groups. Significant progress has been made and agreement reached in several areas. It is expected that such differences as still exist can be resolved, leading to standardization which will be of benefit to all phases of the industry.

Methods of testing hydrometers have been prepared and are now under final review. It is anticipated that this project will be completed at the June meeting.

The subcommittee has been requested by Committee D-2 to prepare specifications for a hydrometer to be used in a new method of test for specific gravity of liquefied petroleum gases.

Subcommittee 19 on Glassware Labora-

tory Apparatus (J. J. Moran, chairman).—The specifications for the glass equipment in the Method of Test for Reduced Pressure Distillation of Petroleum Products (D 1160 - 52 T) have been reviewed, at the request of Research Division VIII, ASTM D-2. New specifications have been drawn up which include some revisions in the apparatus and also complete dimensional requirements for manufacture.

Subcommittees 19 and 21 have collaborated in preparing a proposed E-1 Standard for the equipment used in Method D 95, Test for Water in Petroleum Products and other Bituminous Materials.

Research Division IX, Committee D-2, submitted a draft of a revised Method for Measuring the Color of Petroleum Products to obtain comments on the glassware needed. Subcommittee 19 has approved the specification in the proposed method.

Revised specifications for tar-acid funnels used in Method D 370, Test for Creosote and Method D 453, Test for Tar-Acids in Creosote and Cresote-Coal Tar Solutions, have been prepared and submitted to Subcommittee VI, Committee D-7, for consideration.

Revised specifications for the trap used in Method D 322, Test for Dilution of Crankcase Oils, have been prepared and submitted to Subcommittee Q-1, Technical Committee B, Committee D-2, for consideration.

Subcommittee 21 on Metalware Laboratory Apparatus (E. L. Ruh, chairman) reports progress toward the establishment of "E" Standards covering apparatus for several ASTM Methods. Assistance was given to various working groups on laboratory metalware problems.

The Task Group on Laboratory Ovens held several meetings pointed toward the development of performance requirements for gravity and forced-ventilation type ovens.

Subcommittee 27 on Low-Temperature Testing of Elastomers and Plastics (R. S. Havenhill, chairman).—A final report on Rheological Testing is being completed for publication in the ASTM BULLETIN. The authors of this paper will be C. K. Chatten, W. E. Scoville, Jr., and F. S. Conant.[4] This report will list in tabular form the significant details of nine different types of tests and compare the performance at test temperatures from room to minus 80 F on natural rubber, GR-S, vinyl, and polyethylene stocks. When this work is completed, definite recommendations will be made as to which tests are best suited for low-temperature work.

A report on the brittleness test program is in preparation also for publication in the ASTM BULLETIN. Four low-temperature tests proposed by the ISO/TC 45 on Rubber are under study.

Correlation curves using Bureau of Ships data and Armstrong data show good correlation of Rex, Shore, Pusey and Jones, and ML-1 hardness instruments. This indicates that standard correlation curves can now be prepared.

Subcommittee 28 on Microscopy (F. G. Foster, chairman).—Since microscopy has such a wide scope with many ramifications, endeavor has been made to limit the number of task groups until the present ones are fully established and functioning. To this end there are five task groups on the following subjects: (1) Bibliography, which will be confined to ASTM literature, (2) Methods of Preparation, (3) Standardization and Interchangeability of Elements. This group has been quite active during the past year. Many subjects have come to its attention and a canvass is being made to ascertain which subjects would be most desirable for immediate attention. Cooperation in preparation of Federal Specification for glass slides and covers has been given. (4) Photographic Materials, and (5) Nomenclature.

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[4] Published in ASTM BULLETIN No. 217, Oct., 1956, p. 47 (TP181).

In connection with the June meeting there will be a session on "Non-commercial Accessories and Interesting Techniques." A similar session was held last year and received much favorable comment.

Subcommittee 29 on Microchemical Apparatus (A. Steyermark, chairman).—This subcommittee held its organizational meeting on February 7, 1956, at ASTM Headquarters Besides the details involved in organizing, and developing a program of work, the previous publications in *Analytical Chemistry* of the Committee on Microchemical Apparatus, of the Division of Analytical Chemistry of the American Chemical Society were reviewed with the intention of recommending their acceptance as ASTM Standards. The pieces of apparatus from these previous publications were divided into the following categories: (1) Weighing and Drying, (2) Carbon-Hydrogen, (3) Nitrogen, (4) Sulfur, (5) Halogens, and (6) Alkoxyl.

This report has been submitted to letter ballot of the committee, which consists of 49 voting members; 32 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. R. TOWNSEND,
Chairman.

P. J. SMITH,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee E-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Specifications for:

Weighing and Drying Apparatus for Microchemical Analysis (E 124 – 56 T), and
Apparatus for Determination of Water by Distillation (E 123 – 56 T).

Revision of Tentative Methods of:

Impact Testing of Metallic Materials (E 23 – 47 T), and
Testing and Standardization of Etched-Stem Liquid-in-Glass Thermometers (E 77 – 52 T).

Tentative Revision of Standard Specifications for:

ASTM Thermometers (E 1 – 56 T).

The new Specifications E 124 were accepted by the Standards Committee on October 18, 1956 and appear in the 1956 Supplement to Book of ASTM Standards, Parts 3, 4, 5 and 6, and in the 1956 Book of ASTM Methods for Chemical Analysis of Metals; on November 7, 1956 the new Specifications E 123 were accepted which appear in Parts 3, 4, 5 and 6 of the 1956 Supplement, together with the revision of Methods E 23 which appear in Part 2 of the Supplement; on November 15, 1956 the revisions of Methods E 77 were accepted which appear in Part 5 of the 1956 Supplement, as well as the tentative revision of Specifications E 1 which appears in Parts 3, 4 and 5 of the Supplement.

REPORT OF COMMITTEE E-2

ON

EMISSION SPECTROSCOPY*

Committee E-2 on Emission Spectroscopy held two meetings during the year: in Atlantic City, N. J., on June 30, 1955, and in Pittsburgh, Pa., on February 29, 1956. Meetings of the Executive Subcommittee were held in Atlantic City, N. J., on June 27, 1955; in Philadelphia, Pa., on October 20, 1955; and in Pittsburgh, Pa., on February 29, 1956.

A round-table discussion on X-ray spectroscopy was co-sponsored by the committee with The Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy at Pittsburgh, Pa., on February 29, 1956.

The committee consists of 170 members, representing an increase of six members since the last annual meeting.

The officers elected for the ensuing term of two years are as follows:

Chairman, D. L. Fry.

Vice-Chairman, R. E. Michaelis.

Secretary, Cyrus Feldman.

The committee prepared the Report on Standard Samples and Related Materials for Spectrochemical Analysis—1955 which has been published as *ASTM STP No. 58 C.*

SUGGESTED METHODS TO BE PUBLISHED AS INFORMATION

The committee recommends that the following two suggested methods be published as information.[1]

Suggested Methods for:

Spectrochemical Analysis of Aluminum and its Alloys by the Pin-to-Pin Spark Technique, and

Spectrochemical Analysis of Glass for Alkali Elements by Flame Photometry.

NEW TENTATIVES

The committee recommends that the following method and recommended practices be accepted for publication as tentative:[2]

Tentative Method for:

Spectrochemical Analysis of Pig Lead by the Point-to-Plane Spark Technique.

This method has grown out of Suggested Method E-2 SM 6-3 of similar title, which appears in the 1953 Book of ASTM Methods for Emission Spectrochemical Analysis. The method was tested cooperatively by five laboratories, acting as a task group within Subcommittee VI, and the supporting data are appended hereto.[3]

Tentative Recommended Practices for:

Photographic Processing in Spectrochemical Analysis,[4] and

Photographic Photometry in Spectrochemical Analysis.[4]

These practices were published as Suggested Practices E-2 SM 2-1 and E-2 SM 2-2, respectively, in the 1953 Book of ASTM Methods for Emission Spectrochemical Analysis. Before publication as tentative, the practices for photographic processing will be editorially revised to clarify their general applicability to other materials in addition to the specific products used as examples.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] Available separately on request.

[2] The new tentatives appear in the 1956 Book of ASTM Methods for Chemical Analysis of Metals.

[3] See p. 382.

[4] "Methods for Emission Spectrochemical Analysis," Am. Soc. Testing Mats., pp. 6 and 15 (1953).

TENTATIVES CONTINUED WITHOUT REVISION

The following tentative methods, which have stood for two years without revision, were reviewed by the appropriate subcommittees, and the committee recommends that they be continued as tentative without revision:

Tentative Method for Spectrochemical Analysis of:

Zinc-Base Alloys and High-Grade Zinc by the Solution - D-C Arc Technique (**E 27–53 T**),
Tin Alloys for Minor Constituents and Impurities (**E 51 – 43 T**), and
Aluminum and Aluminum-Base Alloys by the Point-to-Plane Spark Technique (**E 101 – 53 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Apparatus and Equipment (R. G. Russell, chairman).—Group 1 on Testing of Photographic Emulsions is reviewing a report on spectrographic emulsions. Group 3 on Description and Specification of Spectrographic Equipment is preparing a report on source units and spectrographs.

Subcommittee II on Fundamental Methods (R. W. Smith, chairman).—Group 1 on Specification of Spectrographic Sources is studying the definition and differentiation of source units with the object of formulating a uniform acceptable terminology for use in writing methods. Group 2 on Precision and Accuracy assists the subcommittees in the design of experiments for evaluating methods and is preparing directions for specifying precision and accuracy.

Group 3 on Basic Practices in Photographic Photometry recommended that the Suggested Practices for Photographic Photometry (E-2 SM 2-2) be published as tentative, and Group 4 on Basic Practices in Photographic Processing made the same recommendation for Suggested Practices for Photographic Processing (E-2 SM 2-1), referred to earlier in the report.

Groups 5 and 7 on Basic Calculations in Spectrochemical Analysis are reviewing Suggested Methods for Spectrochemical Calculations. Group 6 on Semiquantitative Analysis has prepared a paper entitled "Report of Progress in Obtaining a Universal Method of Spectrochemical Analysis for ASTM"[6] which has been submitted for publication in the ASTM BULLETIN.

Group 8 on Flame Photometry is preparing a proposed tentative method covering basic practices in flame photometry. Questionnaires were mailed to workers in the medical, cement, petroleum, and ceramics fields and the replies analyzed. Group 9 on X-ray Spectrography organized the meeting on X-ray Spectroscopy held at Pittsburgh, Pa., on February 29, 1956. A questionnaire was distributed to ascertain requirements for writing a basic practice in X-ray spectroscopy.

Subcommittee III, Editorial (K. O. Stevenson, chairman).—The Editorial Group reviewed several methods submitted from other subcommittees and prepared manuscripts for the Regulations of the committee and the Recommendations on the Form of Methods. The Nomenclature Group is considering definitions of terms for inclusion in a proposed tentative on spectroscopy nomenclature.

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[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[6] Published in ASTM BULLETIN No. 216, Sept., 1956, p. 29.

Subcommittee IV on Standards and Pure Materials (G. A. Nahstoll, chairman).—The Task Group on Standards Report (R. E. Michaelis, chairman) prepared the Report on Standard Samples and Related Materials for Spectrochemical Analysis which has been published as *ASTM STP No. 58C*. The Group on Standard Samples has continued its cooperation with the National Bureau of Standards. The Group on Electrodes has prepared a report on sizes and shapes of graphite electrodes.

Subcommittee V on Copper, Nickel, and Their Alloys (A. W. Young, chairman).—Group 1 on Sampling of Copper-Base Alloys prepared a report which is being used as a basis for writing a method on sampling. Group 2 completed cooperative testing on Suggested Method for Spectrochemical Analysis of Nickel Alloys by the Powder-D-C Arc Technique (E-2 SM 5-1) in connection with plans to propose this method for publication as tentative. Group 3 on Standard Samples is cooperating with the National Bureau of Standards on preparation of a series of standards. Group 4 on Solution Methods was formed to develop a solution method for the analysis of copper alloys. A method for nickel-base, high-temperature alloys is being reviewed by the subcommittee.

Subcommittee VI on Lead, Tin, Antimony, Bismuth, and Their Alloys (E. J. Dunn, Jr., chairman).—The Suggested Method for Spectrochemical Analysis of Pig Lead by the Point-to-Plane Spark Technique (E-2 SM 6-3) was revised and recommended for publication as tentative as mentioned earlier in the report. The Suggested Method for Spectrochemical Analysis of Tin-Base Alloys by the Dry Powder - D-C Arc Technique (E-2 SM 6-4) will be recommended for publication as tentative upon completion of cooperative testing, and after revision, the Suggested Method for Spectrochemical Analysis of Tin by the Cast Pin – Spark Technique (E-2 SM 6-9) will be recommended for publication as tentative.

Subcommittee VII on Aluminum, Magnesium, and Their Alloys (T. M. Hess, chairman).—The Tentative Method for Spectrochemical Analysis of Aluminum and Aluminum-Base Alloys by the Point-to-Plane Spark Technique (E 101 – 53 T) has been cooperatively tested by four laboratories with the results as appended hereto.[3] A proposed tentative method for Spectrochemical Analysis of Aluminum and Its Alloys by the Point-to-Plane Spark and Intermittent Arc Techniques Using the Production Control Quantometer was prepared and is to be tested cooperatively. Spectrochemical Analysis of Aluminum-Base Alloys by the Dry Powder - D-C Arc Technique (E-2 SM 7-2) is under revision for proposal as a tentative, and Spectrochemical Analysis of Magnesium-Base Alloys by the Cast Pin - Spark and A-C Arc Techniques (E-2 SM 7-3) is being tested by one laboratory preliminary to cooperative testing. Spectrochemical Analysis of Magnesium-Base Alloys by the Point-to-Plane Spark Technique (E-2 SM 7-4) is being cooperatively tested. The Suggested Method for Spectrochemical Analysis of Aluminum and Its Alloys by the Pin-to-Pin Spark Technique (E-2 SM 7-9), referred to earlier in the report, was prepared by the subcommittee.

Subcommittee VIII on Zinc, Cadmium, and Their Alloys (Alan Goldblatt, chairman).—The proposed tentative method for Spectrochemical Analysis of Zinc Die-Casting Metal and High-Purity Zinc by the Cast Pin and Point-to-Plane Techniques is being cooperatively tested.

Subcommittee IX on Ferrous Metals (G. E. Pellissier, chairman).—Group 1 has prepared a proposed tentative method for Spectrochemical Analysis of Carbon and Low-Alloy Steels in the Form of Solid Sections which is undergoing review within the subcommittee. Group 2 is reviewing a proposed tentative method for Spectrochemical Analysis of Carbon and Low-Alloy Steels in the Form of Rods and a suggested procedure for establishing tolerances of a spectrographic method. Group 4 has prepared a proposed tentative method for Direct-Reading Spectrochemical Analysis of Stainless-Type Steels for subcommittee review.

Subcommittee X on Non-Metals (W. J. Poehlman, chairman).—Group 1 on Alkalis is cooperatively testing several methods for the analysis of caustic soda and related materials and is studying a large number of methods including suggested methods already issued, with the purposes of revision, combination, and eventual proposal as tentative. These methods cover caustic soda and a variety of organic and inorganic chemicals. Group 2 on Ceramics and Ores is revising the Suggested Method for Spectrochemical Analysis of Ceramics (E-2 SM 10-9) for proposal as tentative. Group 3 on Slags is preparing three methods including suggested methods on the analysis of slags with a Quantometer, and the photographic analysis of open-hearth slags for lime-silica ratios, and a proposed tentative method for steel-making slags. Group 5 on Flame Photometry prepared the Suggested Method for Spectrochemical Analysis of Glass for Alkali Elements by Flame Photometry (E-2 SM 10-13) referred to previously in the report. Group 6 surveyed available methods of materials associated with the Bureau of Mines and is preparing a suggested method for spectrochemical determination of rare earth elements in cerium minerals. Other groups are working on methods for materials involved in atomic energy, agriculture, biochemistry, petroleum technology, and refractories.

Subcommittee XI on Titanium, Zirconium, and Related Materials (P. A. Leichtle, chairman) reviewed existing suggested methods and is studying several new methods. A porous-cup method already tested cooperatively by a group outside ASTM is under consideration for publication as tentative. Suggested methods for titanium by the solution-spark, rotating disk technique and the point-to-plane spark technique have been prepared and are being reviewed. The group on zirconium and hafnium under the leadership of N. E. Gordon has surveyed the field by means of a questionnaire. Programs have been established for obtaining a series of ZrO_2 standards and a series of metallic zirconium standards.

This report has been submitted to letter ballot of the committee, which consists of 170 members; 97 members returned their ballots, of whom 96 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. R. Churchill,
Chairman.

Cyrus Feldman,
Secretary.

APPENDIX

SUPPORTING DATA RELATING TO METHODS FOR EMISSION SPECTROCHEMICAL ANALYSIS

RESULTS OF COOPERATIVE TESTS ON THE PROPOSED TENTATIVE METHOD FOR SPECTROCHEMICAL ANALYSIS OF PIG LEAD

Supporting data for the Proposed Tentative Method for Spectrochemical Analysis of Pig Lead by the Point-to-Plane Spark Technique are given in Table I.

The method was tested by a task group under the leadership of F. R. Potter, Alcoa Research Laboratories of Aluminum Company of America, with the following companies cooperating:

Vanadium Corporation of America, D. W. Henthorn
Chicago Spectrochemical Laboratories, Alan Goldblatt

TABLE I.—RESULTS OF COOPERATIVE TESTS ON THE PROPOSED TENTATIVE METHOD FOR ANALYSIS OF PIG LEAD.

Element	Average Results of Individual Laboratories, per cent					Grand Average, per cent	Average Percentage Standard Deviation[a]
	A	B	C	D	E		
Copper	0.0029	0.0028	0.0029	0.0031	0.0025	0.00284	2.6
Silver	0.0092	0.0099	0.0092	0.0097	0.0092	0.00944	2.4
Bismuth	0.046	0.045	0.045	0.042	0.0483	0.04526	2.0
Tin	0.011	0.012	0.011	0.012	0.012	0.0116	3.6
Arsenic	0.0095	0.0095	0.0092	0.0094	0.0104	0.0096	3.6
Antimony	0.014	0.014	0.014	0.013	0.015	0.014	5.1
Nickel	0.0016	0.0017	0.0016	...	0.00114	0.00151	3.6
Cadmium	0.0027	0.0025	0.0026	0.0029	0.0035	0.00284	5.5
Tellurium	0.041	0.043	0.041	0.042	0.055	0.0444	5.6

[a] Percentage standard deviation, ν, for each laboratory is calculated as follows:

$$\nu = \frac{100}{\bar{c}} \sqrt{\frac{\Sigma (\bar{c} - c)^2}{n}}$$

where:
$\bar{c}$ = average concentration by laboratory, per cent,
c = individual determination, per cent, and
n = number of determinations.

REPORT ON COOPERATIVE TESTS ON THE TENTATIVE METHOD FOR SPECTROCHEMICAL ANALYSIS OF ALUMINUM (E 101 – 53 T)

The Tentative Method for Spectrochemical Analysis of Aluminum and Aluminum-Base Alloys by the Point-to-Plane Spark Technique (E 101 – 53 T)

Frankford Arsenal, A. L. Jamieson
The Dow Chemical Company, T. M. Hess

Each participant was provided with one unknown and a series of standards for each of three alloy types. Each series of standards included a nominal standard having a composition fairly typical of the alloy under test and sufficient additional standards to prepare analytical curves for all elements to be determined. After establishing analytical curves, the unknown and nominal standard for each alloy were tested six times each (alternately) on each of four days. The analytical curves were adjusted according to the average results for the nominal standards for each day, and the analysis for each test on the unknown determined.

TABLE II.—SUMMARY OF THE ANALYSES OF THE SAMPLES (METHOD E 101 – 53 T).

Alloy Type	Element	Chemical Value, per cent[a]	Spectrochemical Values, per cent			
			Laboratory 1	Laboratory 2	Laboratory 3	Laboratory 4
2024	Copper	4.58	4.55	4.54	4.62	4.52
	Iron	0.34	0.34	0.34	0.34	0.35
	Silicon	0.32	0.35	0.31	0.32	0.31
	Manganese	0.66	0.66	0.66	0.67	0.65
	Magnesium	1.72	1.72	1.68	1.70	1.66
	Zinc	0.067	0.068	0.066	0.068	0.067
	Nickel	0.041	0.047	0.043	0.044	0.042
	Chromium	0.063	0.063	0.064	0.064	0.062
	Titanium	0.041	0.044	0.043	0.042	0.042
5056	Copper	0.052	0.052	0.060	...	0.049
	Iron	0.20	0.20	0.21	0.21	0.21
	Silicon	0.12	0.12	0.12	0.12	0.12
	Manganese	0.10	0.10	0.10	0.10	0.099
	Magnesium	4.84	4.80	4.64	4.75	4.88
	Zinc	0.045	0.049	0.046	0.045	0.046
	Nickel	0.047	0.050	0.045	0.062	0.048
	Chromium	0.12	0.12	0.12	0.12	0.12
	Titanium	0.029	0.030	0.031	0.012	0.030
356	Copper	0.19	0.20	0.19	0.18	0.18
	Iron	0.37	0.38	0.40	0.38	0.39
	Silicon	7.05	6.97	6.96	6.90	6.92
	Manganese	0.12	0.12	0.12	0.12	0.11
	Magnesium	0.32	0.35	0.34	0.35	0.34
	Zinc	0.10	0.12	0.11	0.11	0.11
	Nickel	0.051	...	...	...	...
	Chromium	0.052	...	...	...	...
	Titanium	0.12	0.11	0.12	...	0.11

[a] Values determined by Alcoa Research Laboratories.

Table II shows the alloys tested, the elements determined, and the average spectrochemical results for each laboratory. Also shown for purposes of comparison is a chemical analysis of each unknown.

A condensed summary of a statistical analysis of the data for the most important determinations is shown in Table III. The coefficients of variation within days were based on the tests on both unknowns and standards, because this made available a greater number of degrees of freedom and because there was no significant difference in observed variability between unknowns and standards. The conventional *"F"* test was employed to determine whether there was any significant variation among results obtained on different days other than that to be expected from the variation within days. For the same purpose, the observed coefficient of variation in daily means is compared with the values calculated from the variation within days.

From the results of these cooperative tests, it is concluded that with sufficiently

TABLE III.—STATISTICAL ANALYSIS OF A PORTION OF THE TEST DATA (METHOD E 101 – 53 T).

Alloy and Element	Laboratory	Coefficient of Variation Within Days	F[a]	Coefficient of Variation Among Daily Means	
				Observed	Calculated[b]
2024, Copper at 4.5 per cent	1	1.17	2.42	0.77	0.68
	2	2.08	1.34	1.04	1.20
	3	2.86	1.52	1.44	1.65
	4	1.02	0.12	0.18	0.59
2024, Manganese at 0.7 per cent	1	2.04	5.58	2.00	1.18
	2	2.16	6.02	1.95	1.25
	3	2.38	0.62	0.86	1.37
	4	1.37	3.33	1.26	0.79
2024, Magnesium at 1.7 per cent	1	1.80	1.21	1.00	1.04
	2	2.26	1.79	1.46	1.30
	3	2.28	4.07	1.76	1.32
	4	1.60	0.37	0.35	0.92
5056, Magnesium at 4.8 per cent	1	2.82	0.78	1.08	1.63
	2	1.73	3.84	1.22	1.00
	3	2.17	3.69	1.81	1.25
	4	1.26	0.97	0.77	0.73
356, Silicon at 7.0 per cent	1	1.87	0.89	0.98	1.08
	2	1.76	1.15	0.84	1.02
	3	1.92	1.07	0.98	1.11
	4	1.25	0.04	0.08	0.72
Degrees of freedom		40	3/20[c]	3	40

[a] F = ratio of variance among days to variance within days. If there is no variation among days other than that attributable to the variation within days,

Probability of $F > 2.38 = 0.10$
Probability of $F > 3.10 = 0.05$
Probability of $F > 4.94 = .01$

[b] Calculated from observed variation within days.

[c] Degrees of freedom among days = 4 − 1 = 3
Degrees of freedom within days = 24 − 4 = 20

uniform samples, this method can be expected to give coefficients of variation ranging from 1 per cent to 3 per cent. It is further concluded that there are no significant biases among the results of different laboratories analyzing the same samples with the same standards, nor between the results of the same laboratory obtained at different times. In the judgment of the task group, the alloys tested are representative of the general field of aluminum alloys. While only the data representing higher concentrations were formally analyzed statistically, the results for all elements tested showed satisfactory agreement among laboratories and with the chemically determined values.

Variabilities in sampling practices are not included in these tests since only one unknown of each alloy was tested. The unknowns were especially selected to provide a large amount of uniform material in order to permit all participants to work on the same samples. It is planned to study variables in sampling in subsequent tests on other methods employing the same types of samples as are conventionally used in this method.

REPORT OF COMMITTEE E-3
ON
CHEMICAL ANALYSIS OF METALS*

Committee E-3 on Chemical Analysis of Metals held one meeting during the year: on June 29, 1955, in Atlantic City, N. J., in conjunction with the Annual Meeting of the Society.

A new edition of the Book of ASTM Methods of Chemical Analysis of Metals, scheduled to appear the latter part of 1956, will include a number of new methods and revisions of existing methods. This edition covers adequately the chemical requirements of the majority of ASTM specifications for metals.

Cooperation with other ASTM committees has continued; for example, with Committee B-9 on Metal Powders and Metal Powder Products, with Committee B-4 on the analysis for gases in nickel, and with Committee A-1 on the accuracy and precision of methods for analysis of ferrous metals.

Major milestones include the preparation of methods for the analysis of titanium and its alloys, the analysis of ferro-alloys, a positive interest in methods for gases in nickel and steels, and the re-establishment of annual symposia on some timely analytical subject.

Other projects illustrating the committee's broadening scope of activities are the consideration of methods for ore analysis, methods for impurities in germanium, and the possible recommendation of nationally and internationally known standard samples for analytical purposes.

The officers elected for the ensuing term of two years are as follows:

Chairman, Arba Thomas.
Vice-Chairman, C. Zischkau.
Secretary, H. Kirtchik.

NEW TENTATIVES

The committee recommends that the following methods be accepted for publication as tentative as appended hereto:[1]

Methods for Chemical Analysis of:
Titanium and Titanium-Base Alloys,
Nickel-Chromium and Nickel-Chromium Iron Alloys
Copper - Tellurium Alloys,
Chromium - Copper Alloys,
Slab Zinc (Spelter), and

Methods for Determination of:
Phosphorus in High-Phosphorus Brazing Alloys.

REVISIONS OF TENTATIVES

The committee recommends that the following five methods be revised as indicated and continued as tentative:

Tentative Photometric Method for Determination of Manganese in Steel (E 30 – 54 T):

Revise by the addition of methods of analysis for magnesium, for nickel, and for columbium and tantalum, as appended hereto.[1]

Tentative Photometric Methods for Chemical Analysis of Aluminum and Aluminum-Base Alloys (E 34 – 54 T):

Revise by the adoption as standard of the present Sections 6 to 56, as mentioned later in this report, and by the addition of a new method for the determination of magnesium. The revised methods are appended hereto.[1]

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* **Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.**

[1] The new and revised tentatives appear in the 1956 Book of ASTM Methods for Chemical Analysis of Metals.

Tentative Methods for Chemical Analysis of White Metal Bearing Alloys (E 57 – 50 T):[2]

Section 15.—Change to read as follows:

15. (*a*) *Standard Potassium Bromate Solution* (*0.01N or 0.05 N*).—Reagent No. 9.

(*b*) *Methyl Orange Indicator Solution* (*1 g. per l.*).—Reagent No. 118.

Section 16 (*a*) *and* (*b*).—Change to read as follows:

16. (*a*) Transfer 0.500 to 2.000 g. of the sample (the amount depending on the antimony content) to a 500-ml. Erlenmeyer flask. To the flask, and to a second flask to be carried through the entire procedure as a blank, add 15 ml. of H_2SO_4 and about 7 g. of $KHSO_4$ or $K_2S_2O_7$. Heat to decompose the sample, avoiding too high temperatures during the initial heating; otherwise the sample may melt and incomplete decomposition may result. When decomposition is complete, heat vigorously over an open flame to expel or wash down the sulfur on the walls of the flask.

(*b*) Cool, add 10 ml. of water, shake to hydrate the H_2SO_4, and again cool. Add three or four granules of silicon carbide to prevent bumping. Add 75 ml. of HCl and 1 g. of Na_2SO_3. Heat the solution to gentle boiling and boil without cover until the volume has been reduced to 60 ± 5 ml. Dilute to 300 ml. with boiling water, bubble a fairly vigorous stream of air through the solution for 1 min., and titrate the hot solution with the appropriate $KBrO_3$ solution, using methyl orange as the indicator, which should preferably be added near the end of the titration.

Tentative Photometric Methods for the Chemical Analysis of Lead, Tin, Antimony, and Their Alloys (E 87 – 50 T):[2]

Section 18.—Change to read as follows:

18. Gold, the platinum group metals, ferric iron, and, to a lesser extent, antimony interfere. The method provides for the removal of the interference due to iron and antimony.

Section 19.—Change Paragraphs (*b*) and (*c*) to read as follows:

(*b*) *Standard Copper Solution* (*1 ml. = 0.1 mg. Cu*).—Dissolve 0.1000 g. of copper in 3 ml. of HNO_3 by gentle heat in a 125-ml. conical flask. Add 10 ml. of $HClO_4$ and heat to copious fumes of $HClO_4$ to expel HNO_3. Cool, add 10 ml. of water, transfer to a 1-liter volumetric flask, and dilute to the mark.

(*c*) *Test Lead.*—Finely granulated test lead containing under 0.0001 per cent of copper and under 0.001 per cent of iron or nickel.[16, 20]

Section 20.—Change Paragraphs (*a*) and (*b*) to read as follows:

(*a*) *Calibration Solutions:*

(*1*) Transfer 0.5, 1.0, 2.0, 3.0, 4.0, 6.0, and 8.0 ml. of copper solution (1 ml. = 0.1 mg. Cu) to 125-ml. conical flasks. Add 2 ml. of $HClO_4$ and dilute to 40 ml.

(*2*) Add 1 g. of test lead to each sample, cover, and boil at a moderate rate for 15 min. to displace all the copper. Cool somewhat, remove the solution by decantation, and wash once with water—decanting thoroughly. Add 10 ml. of $HBr - Br_2$ mixture to the flask, cover, and heat gently to dissolve the metal. Boil to expel the excess bromine. Cool to room temperature. Transfer 10 ml. of H_3PO_4 plus 1 drop (0.05 ml.) of $HBr - Br_2$ mixture to a dry, 25-ml. volumetric flask.[15] Transfer the sample solution to the volumetric flask, washing with a few milliliters of HBr. Dilute to the mark with HBr and mix. Proceed in accordance with Paragraph (*c*).

(*b*) *Reference Solution.*—Transfer 40 ml. of water plus 2 ml. of $HClO_4$ to a 125-ml. conical flask and continue in accordance with Paragraphs (*a*)(*2*) and (*c*).

Section 21 (*a*).—Change to read as follows:

(*a*) *Sample Solution:*

(*1*) Depending on the copper content, transfer up to 2.00 g. of the sample, from which adventitious iron has been removed, to a 125-ml. conical flask. Add 10 ml. of HNO_3 (1:3), cover, and heat gently until the sample is dissolved. Add 5 ml. of $HClO_4$ and boil to a volume of 2 ml.

(*2*) Add 40 ml. of water and 1 g. of test lead and continue in accordance with Section 20(*a*)(*2*).[16]

Section 24.—Change the heading preceding this section to read: "Procedure for Pig Lead and Lead- and Tin-Base Alloys Containing Small Amounts of Iron and Less Than 1 per cent of Antimony[20, 21]."

Change this section to read as follows:

24. (*a*) *Sample Solution.*—Depending on the copper content, transfer up to 1.00 g. of the

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[2] 1950 Book of ASTM Methods for Chemical Analysis of Metals.

sample, from which adventitious iron has been removed, to a 150-ml. beaker. Add 10 ml. of $HBr - Br_2$ mixture. Cover and heat gently, avoiding excessive loss of bromine, until dissolution of the sample is complete. When analyzing high-tin alloys it may be necessary to add a few extra drops of bromine to insure complete dissolution of the sample and oxidation of the tin to the stannic state. When dissolution is complete boil gently until all the bromine has been removed.[21] Cool to room temperature and transfer to a 25-ml. volumetric flask containing 10 ml. of H_3PO_4 plus 1 drop (0.05ml.) of $HBr - Br_2$ mixture, washing with a few milliliters of HBr. Dilute to the mark with HBr and mix.

(*b*) Complete the determination in accordance with Section 21 (*b*) to (*d*).

Footnotes 15, 16, 17a, and 22.—Delete these footnotes, renumbering the subsequent footnotes accordingly.

Footnote 24.—Change to read as follows:

[21] High results for copper are obtained if excessive amounts of bromine or antimony are present at the time of photometric measurements.

The committee had submitted revisions of Sections 11 to 13 of Methods 887 to the Administrative Committee on Standards in 1954. Although these had been accepted by the Standards Committee as given in the 1954 annual report, the committee now recommends its revision not be made.

Tentative Photometric Methods for the Chemical Analysis of Electronic Nickel (**E 107 – 54 T**):[2] Revise by the addition of methods for the determination of aluminum, carbon, hydrogen, nitrogen, and oxygen, as appended hereto.[1]

REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommends that the Standard Methods for Chemical Analysis of Zinc-Base Die-Casting Alloys (**E 47 – 45**)[2] be revised as appended hereto[1] and reverted to tentative status. The revisions include the incorporation of the methods for determination of aluminum now published under the designation E 47 – 50 T.[2]

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following nine tentative methods be approved for reference to letter ballot of the Society for adoption as standard without change:

Tentative Photometric Methods for:

Chemical Analysis of Aluminum and Aluminum-Base Alloys (**E 34 – 54 T**), with the exception of the method for silicon covered in Sections 57 to 64, which is being retained as tentative.

Chemical Analysis of Magnesium and Magnesium-Base Alloys (**E 35 – 50 T**),[2]

Determination of Cobalt in Nickel (**E 39 – 49 T**),[2] and

Chemical Analysis of Copper and Copper-Base Alloys (**E 62 – 50 T**).[2]

Tentative Methods for Chemical Analysis of:

Pig Lead (**E 37 – 50 T**),[2]

Lead- and Tin-Base Solder Metal (**E 46 – 50 T**),[2]

Copper-Nickel and Copper-Nickel-Zinc Alloys (**E 75 – 50 T**),[2] and

Nickel-Copper Alloys (**E 76 – 50 T**).[2]

Tentative Method for Polarographic Determination of:

Lead and Cadmium in Zinc (**E 68 – 46 T**).[2]

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions of the following standards as indicated, and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Methods for Chemical Analysis of Steel, Cast Iron, Open-Hearth Iron, and Wrought Iron (**E 30 – 52**):

Section 6.—Change the heading preceding this section to read: "Procedure for Carbon and High-Silicon Steels Containing Under 0.1 per cent Sulfur."

In Paragraph (*a*), line 3, change "1 to 3 g" to "1 to 5 g."

Sections 20, 28, 35, and 43.—Change the headings preceding these sections to read: "Procedure for Cast Iron and High-Silicon Steels."

Footnote. 21—Delete this footnote,

renumbering Footnotes 22 to 40*b* accordingly.

Section 51.—Change the heading preceding this section to read: "Procedure for Open-Hearth Iron, Cast Iron, Wrought Iron, and High-Silicon Steels."

Section 55.—Change the heading preceding this section to read: "Procedure for Alloy and High-Silicon Steels."

Sections 62 and 68.—Change the headings preceding these sections to read: "Procedure for Carbon and High-Silicon Steels."

Section 79.—Change the heading preceding this section to read: "Procedure for Cast Iron and High-Silicon Steel."

Change Paragraph (*b*) to read:

(*b*) Wash the residue with hot water and ignite in platinum at a temperature not exceeding 550 C. Add 2 ml of H_2SO_4 (1:1) and 3 to 5 ml of HF, evaporate to dense white fumes, cool and add to the main filtrate. Dilute the filtrate to 500 ml with hot water, and complete the determination as described in Section 75.

Section 86.—Change the heading preceding this section to read: "Procedure for Cast Iron and High-Silicon Steel."

Add the following sentences to Paragraph (*a*): "Ignite in platinum at a temperature not exceeding 550 C. Add 2 ml of H_2SO_4 (1:1) and 3 to 5 ml of HF, evaporate to dense white fumes, and cool. Add 10 ml of water and filter into the main filtrate, washing the residue with hot water."

Section 93.—Change the heading preceding this section to read: "Procedure for Cast Iron and High-Silicon Steels."

Section 108.—Change the heading preceding this section to read: "Procedure for Cast Iron Containing Under 0.15 per cent Chromium, Open-Hearth Iron, Wrought Iron, and High-Silicon Steels."

Section 118.—Change the heading preceding this section to read: "Procedure for Cast Iron Containing Under 0.05 per cent Vanadium, Open-Hearth Iron, Wrought Iron, and High-Silicon Steels."

Section 130 (*b*).—After the first sentence, insert the following new sentences: "In the case of high-silicon steel, add 0.5 ml of HF before evaporating to white fumes. Cool, wash down the sides of the flask with water, and again evaporate to white fumes."

Standard Methods for Chemical Analysis of Nickel-Chromium and Nickel-Chromium-Iron Alloys (E 38 – 48):[2]

Section 25.—Add a new Paragraph (*b*) to read as follows:

(*b*) Alternatively, transfer 1 g of the sample to a 400-ml beaker, add 50 ml of HNO_3 - HCl mixture, cover, and heat until solution is complete. Add 25 ml of $HClO_4$ (70 per cent) and evaporate to fumes; boil gently 15 min and cool. Wash cover and add 75 ml of hot water. Dissolve salts and filter into a 200-ml volumetric flask, using a small amount of filter pulp to aid filtration. Wash thoroughly with hot water, remove the flask, and replace with the original beaker. Wash twice with hot HCl (2:5). Wash twice with hot water. Dilute the contents of the flask to the mark and mix. Reserve for the determination of manganese and copper.

Renumber the present Paragraphs (*b*) to (*e*) accordingly.

WITHDRAWAL OF TENTATIVE

In view of the incorporation of the procedure for iron by the thiocyanate method into the new Tentative Methods for Chemical Analysis of Slab Zinc, mentioned earlier in the report, the committee recommends the withdrawal as a separate designation of the Tentative Photometric Method for Determination of Iron in Slab Zinc (Spelter) **(E 64 – 50 T)**.[2]

REAPPROVAL OF STANDARD

The committee recommends the reapproval as standard of the Methods for Chemical Analysis of Slab Zinc (Spelter) **(E 40 – 45)**.[2]

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that pending further study and experience in their use, the following tentatives be continued without revision:

Methods for Chemical Analysis of:

Ferro-Alloys (**E 31 – 54 T**),
Sulfur in Special Brasses and Bronzes (**E 54 – 50 T**),
Antimony Metal (**E 86 – 50 T**), and
Copper-Beryllium Alloys (**E 106 – 54 T**).

Methods of Sampling:

Non-Ferrous Metals and Alloys in Cast Form for Determination of Chemical Composition (**E 88 – 50 T**).

Recommended Practice for:

Photometric Methods for Chemical Analysis of Metals (**E 60 – 50 T**).

Supporting data for the preceding recommendations are given in the Appendix.[3]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Editorial Subcommittee (J. L. Hague, chairman).—A style manual for methods of the committee will be prepared after the adoption of a Society style manual for chemical methods in general being prepared by Subcommittee 15 of Committee E-1.

Division F on Ferrous Metals (J. J. Furey, chairman).—Based upon work at the National Bureau of Standards, the method for analysis of ferroboron is being rewritten. A task group will continue work on this project, which contemplates providing a standard sample of ferroboron as well as a method of analysis.

Suitable samples together with suggested methods have been distributed to the respective task groups for work on (1) determination of aluminum in stainless type alloy steels, and (2) determination of cobalt in high cobalt jet-type steels.

The division prepared the methods for determination of columbium and tantalum in stainless steels, and determination of magnesium in nodular iron which are recommended earlier in the report for inclusion in Tentative Methods E 30 – 54 T.

Much work has been done on a photometric method for nickel in steels, but further work is needed to apply corrections for interferences.

Additional work on determination of sulfur by direct combustion will begin in the near future. A task group using the various types of induction furnaces now available has been organized; some difficulty had been experienced in getting boats with a uniformly low blank but this has recently been rectified.

The division prepared the modification of Standard Methods E 30 – 52 to include high-silicon steels, referred to earlier in the report.

A request for a method for determination of hydrogen in steel has led to the formation of a task group to study the problem. Since sampling plays a vital part in this analysis, the task group will also be charged with the responsibility for developing a satisfactory sampling procedure.

Division M on Miscellaneous Metals (R. M. Fowler, chairman) has developed analytical methods for titanium and its alloys in cooperation with the Metallurgical Advisory Committee on Titanium of Watertown Arsenal, and the methods are being recommended for publication as tentative as mentioned earlier in this report. Included are methods for aluminum, chromium, iron, manganese, molybdenum, nitrogen, tin, and vanadium.

A task group has developed improved methods for the determination of copper and manganese in electrical heating alloys and also a modification of the present method (E 38 – 48) for silicon in these materials as referred to earlier in the report.

As a joint project with Committee F-1

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[3] See p. 391.

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

on Materials for Electron Tubes and Semiconductor Devices, work has been done on methods for carbon, oxygen, hydrogen, and nitrogen in electronic nickel and similar materials. These methods are being recommended as a revision of Method E 107 - 54 T, as mentioned earlier in the report.

Division S on Sampling of Metals (A. C. Holler, chairman):

Subcommittee S-1 on Ferrous Metals (J. B. Armstrong, chairman).—A task group is studying certain aspects of the sampling of pig iron and the possible revision of the Standard Method of Sampling Steel, Cast Iron, Open-Hearth Iron, and Wrought Iron (E 59 - 45).

Sampling procedures for tubular products for chemical analysis are being studied by a task group in cooperation with Committee A-1 on Steel.

Subcommittee S-2 on Non-Ferrous Metals (R. L. Vitek, chairman).—Methods for the sampling of titanium sponge are being prepared jointly with Subcommittee VIII on Miscellaneous Refined Metals and Alloys of Committee B-2.

Division N on Non-Ferrous Alloys (R. G. Ernst, chairman) has prepared the new methods for analysis of copper-tellurium alloys, chromium-copper alloys, slab zinc (spelter), and high-phosphorus brazing alloys mentioned earlier in the report. The division also prepared the revisions of Methods E 34 - 54 T, E 47 - 45, E 57 - 50 T, and E 87 - 54 T. The revisions of Methods E 107 - 54 T were prepared jointly with Division M.

Work is under way on (1) determination of antimony in pig lead and fire refined copper with rhodamine, (2) use of neo-cuproine as a general photometric reagent for determination of copper, (3) determination of zirconium in magnesium-base alloys, and (4) determination of zinc in aluminum-base alloys by means of solvent extraction, followed by versene titration.

Division G on General Analytical Methods (H. F. Beeghly, chairman) is responsible for planning annual symposia of timely interest to Committee E-3 and other ASTM committees interested in chemical analysis. The first of these on "Ion Exchange and Chromatography in Analytical Chemistry"[5] will be held at the 1956 Annual Meeting.

Subcommittee G-1 on Reagents, G-2 on Apparatus, and G-3 on Common Procedures have been reviewing methods for publication in the new Book of ASTM Methods for the Chemical Analysis of Metals.

Subcommittee G-3 is reviewing methods for determination of phosphorus with the object of developing a common procedure for this element.

Subcommittee G-4 on Statistics has compiled and distributed a suggested procedure for conducting cooperative interlaboratory tests on standard procedures.

This report has been submitted to letter ballot of the committee, which consists of 121 members; 65 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

ARBA THOMAS,
Chairman.

H. KIRTCHIK,
Secretary.

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[5] Issued as separate publication *ASTM STP No. 195.*

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee E-3 presented to the Society through the Administrative Committee on Standards the recommendation that the Tentative Methods for Chemical Analysis of Steel, Cast Iron, Open-Hearth Iron, and Wrought Iron (E 30 - 51 T) be revised. This recommendation was accepted by the Standards Committee on September 5, 1956, and the revised tentative methods appear in the 1956 Book of ASTM Methods for Chemical Analysis of Metals.

APPENDIX

SUPPORTING DATA RELATING TO NEW AND REVISED METHODS FOR CHEMICAL ANALYSIS OF METALS

Supporting data for the various new and revised methods for chemical analysis of metals included in the 1956 report of Committee E-3 are given in the following Tables I to XIII.

TABLE I.—RESULTS OF COOPERATIVE TESTS ON THE PROPOSED TENTATIVE METHODS FOR CHEMICAL ANALYSIS OF TITANIUM AND TITANIUM-BASE ALLOYS.

Aluminum by the 8-Hydroxyquinoline Gravimetric Method

(Method as written, all interfering elements, synthetic samples.)

Laboratory No.	Aluminum Present, per cent	Aluminum Found, per cent
1	0.05	0.053
2		0.045
3		0.049
1	0.10	0.10
2		0.12
3		0.10
1	0.25	0.25
2		0.25
3		0.26
1	0.50	0.52
2		0.51
3		0.52
1	5.0	4.95
2		4.99

Chromium by the Persulfate Oxidation Method

(Results marked with an asterisk (*) were determined by potentiometric titration.)

Laboratory No.	Sample	Chromium, per cent		
		Max	Min	Avg
1	WA-2	5.18	5.15	5.16
2		5.21	5.14	5.18
3		5.19	5.11	5.16
4		5.16	5.12	5.15
5		5.1	5.0	5.0*
1	WA-5	9.98	9.94	9.96
2		10.05	9.97	10.01
3		10.04	9.94	9.97
4		9.84	9.75	9.78
5		10.0	9.7	9.8*
1	WA-7	2.12	2.08	2.11
2		2.16	2.11	2.13
3		2.15	2.07	2.12
4		2.16	2.05	2.10
5		2.1	2.1	2.1*
1	WA-12	2.78	2.74	2.76
2		2.80	2.76	2.78
3		2.82	2.78	2.81
4		2.72	2.69	2.71
5		2.7	2.7	2.7*

Iron by the Sulfide Separation - Dichromate Titration Method

Laboratory No.	Sample	Iron, per cent		
		Max	Min	Avg
1	WA-20	0.36	0.35	0.355
2		0.38	0.36	0.37
3		0.19	0.39	0.26
4		0.343	0.339	0.341
1	WA-21	4.94	4.62	4.81
2		4.91	4.84	4.88
3		4.72	4.56	4.66
4		4.970	4.924	4.95
1	WA-29	0.13	0.10	0.118
2		0.11	0.09	0.10
3		0.098	0.094	0.096
1	WA-35	2.30	2.26	2.28
2		2.29	2.27	2.28
3		2.22	2.14	2.20
4		2.363	2.305	2.32
1	WA-36	1.72	1.68	1.70
2		1.65	1.57	1.60
3		1.759	1.681	1.726

Manganese by the Persulfate - Arsenite Method

Laboratory No.	Sample	Manganese, per cent
1	WA-9	3.58, 3.57, 3.59, 3.59, 3.57
2		3.60, 3.58, 3.58, 3.55, 3.55, 3.55, 3.55, 3.55, 3.53, 3.50
3		3.41, 3.46 (preliminary results)
4		3.61, 3.63, 3.63 (preliminary results)
1	WA-10	0.057, 0.054, 0.053
2		0.090, 0.090 (preliminary results)
3		0.068, 0.068, 0.046, 0.049 (preliminary results)
1	WA-11	5.42, 5.34, 5.33, 5.29, 5.24
2		5.22, 5.22, 5.20, 5.20, 5.19, 5.18, 5.15, 5.15, 5.15, 5.15, 5.13, 5.10
3		5.10, 5.09 (preliminary results)
1	WA-34	8.14, 8.14, 8.11, 8.14, 8.14, 8.14, 8.14
2		8.00, 8.10, 8.05, 8.03, 8.13, 8.13, 8.03, 8.03
3		8.35, 8.30, 8.31, 8.30

TABLE I.—RESULTS OF COOPERATIVE TESTS ON THE PROPOSED TENTATIVE METHODS FOR CHEMICAL ANALYSIS OF TITANIUM AND TITANIUM-BASE ALLOYS (*Continued*).

IRON BY THE ORTHO-PHENANTHROLINE (PHOTOMETRIC) METHOD

Determination No.	Sample	Iron, per cent		
		Max	Min	Avg
1	WA-20	0.38	0.35	0.363
2		0.36	0.33	0.345
3		0.36	0.34	0.35
4		0.39	0.35	0.370
5		0.340	0.332	0.336
6		0.376	0.309	0.349
1	WA-21	5.30	4.97	5.16
2		4.95	4.93	4.94
3		4.80	4.68	4.74
4		5.16	5.10	5.07
5		5.00	4.93	4.96
6		4.95	4.84	4.90
1	WA-29	0.104	0.090	0.097
2		0.086	0.085	0.086
3		0.075	0.074	0.065
4		0.101	0.091	0.093
5		0.094	0.086	0.089
1	WA-35	2.54	2.25	2.424
2		2.29	2.26	2.273
3		...	...	2.12
4		2.40	2.33	2.365
5		2.33	2.26	2.290
6		2.28	2.28	2.28
1	WA-36	1.90	1.73	1.79
2		1.71	1.69	1.70
3		1.67	1.62	1.64
4		1.70	1.70	1.70
5		1.84	1.62	1.74
6		1.80	1.68	1.74
7		1.71	1.71	1.71

NITROGEN BY THE DISTILLATION - TITRATION METHOD

(Average of four results.)

Laboratory No.	Nitrogen, per cent					
	Sample RC 130B	Sample Ti 150A	Sample WA-30	Sample WA-31	Sample WA-35	Sample WA-37
1	...	...	0.019	0.039	0.108	0.130
2	0.036	0.082	0.018	0.038	0.108	0.130
3	0.037	0.084	0.020	0.040	0.110	0.134
4	0.036	0.081	...	...	...	...
5	...	...	0.019	0.041	0.107	0.130
6	0.038	0.083	0.020	0.041	0.111	0.131
Averages	0.037	0.082	0.019	0.040	0.109	0.131

MOLYBDENUM BY THE THIOCYANATE (PHOTOMETRIC) METHOD

Laboratory No.	Sample	Molybdenum, per cent
1	WA-4 (50-mg samples)	3.12, 3.18, 3.18 (Beckman)
2		2.98, 2.98 (Klett)
3		3.12, 3.05, 3.04, 3.20
1	WA-6 (50-mg samples)	9.16, 9.23, 9.26, 9.54, 9.38, 9.38
2		9.25, 9.23, 9.25, 9.34, 9.22, 9.25, 9.25 (separate first aliquots)
2		9.28, 9.30, 9.28, 9.24, 9.28, 9.32, 9.28 (same first aliquot)
3		9.14, 9.40, 9.24
1	WA-7 (50-mg samples)	2.06, 1.93, 2.06, 2.20, 2.00, 2.04
2		1.84, 1.84
3		1.84, 1.86, 1.84, 1.86
1	WA-4 (using a 1-g sample and aliquoting)	3.10, 3.06, 3.06, 2.96, 3.04
	WA-6	9.00, 9.10, 9.10
	WA-7	1.80, 1.80, 1.80

MANGANESE BY THE PERIODATE OXIDATION (PHOTOMETRIC) METHOD

Laboratory No.	Sample	Manganese, per cent
1	WA-9	3.45, 3.51, 3.55, 3.53, 3.53, 3.51, 3.50, 3.54, 3.54
1	WA-10	0.070, 0.072, 0.068, 0.070, 0 070, 0.071, 0.070, 0.068, 0.068, 0.068
2		0.058, 0.058, 0.056, 0.056
3		0.050, 0.053, 0.049, 0.045, 0.047, 0.050, 0.055, 0.048, 0.046, 0.048
4		0.056, 0.056
1	WA-11	5.08, 5.28, 5.24, 5.16, 5.20, 5.24, 5.20, 5.28, 5.24, 5.28, 5.20, 5.08
1	WA-34	8.10, 8.10, 8.12, 8.10, 8.10, 8.00, 8.08, 8.08, 8.08, 8.20, 7.98

MOLYBDENUM BY THE SULFIDE - PERMANGANATE TITRATION METHOD

(Since this was a one-man task force, there is no comparative data on the method. However, the method is essentially the same as the method for molybdenum in ferromolybdenum which is published as Sections 73 to 75 of the Standard Methods for Chemical Analysis of Ferro-Alloys (E 31 – 47).)

TABLE I.—RESULTS OF COOPERATIVE TESTS ON THE PROPOSED TENTATIVE METHODS FOR CHEMICAL ANALYSIS OF TITANIUM AND TITANIUM-BASE ALLOYS (*Continued*).

TIN BY THE IODIMETRIC TITRATION METHOD

Laboratory No.	Titrant	Tin, per cent						Variations
		Sample WA-44			Sample WA-45			
1..	0.02 *N* KIO_3	2.61	2.62	2.63	4.22	4.26	4.24	
		2.63	2.63	2.62	4.24	4.21	4.24	
		2.62	2.57	2.64	4.25	4.20	4.21	Sulfide separation
	0.097 *N* iodine	2.58	2.58	2.58	4.22	4.22	4.16	
		2.60	2.60	2.58	4.19	4.22	4.22	
2...	0.01 *N* iodine	2.64	2.63	2.65	4.32	4.34	4.37	
		2.63	2.62	2.63	4.33	4.31	4.24	
		2.66	2.66	2.66				2 per cent V added
		2.67	2.66	2.66				3 per cent V added
		2.94	2.91	3.11				6 per cent V added
		2.58	2.60	2.60				6 per cent Mo added
		2.66	2.65	2.66				0.5 per cent W added
3...	0.02 *N* iodine	2.65	2.65	2.65	4.31	4.31	4.30	
		2.64	2.66		4.35	4.40		
		2.67	2.63		4.29	4.27		Reduced 10 g Armco iron
		2.77	2.84		4.33	4.49		Reduced 5 g Armco iron
		2.67	2.70		4.33	4.32		Reduced 10 g Plast iron
4...	0.02 *N* iodine	2.69	2.70	2.70	4.17	4.24	4.26	Reduced with nickel
		2.69	2.65	2.68	4.24	4.24	4.27	Reduced with nickel
5...	0.02 *N* iodine	2.62	2.64	2.66	4.35	4.38	4.39	Sulfide separation

VANADIUM BY THE PERMANGANATE OXIDATION - FERROUS SULFATE TITRATION METHOD

Laboratory No.	Sample	Vanadium, per cent		
		Max	Min	Avg
1.......	WA-14	2.56	2.53	2.55
2.......		2.71	2.67	2.69
3.......		2.68	2.50	2.60
4.......		2.68	2.65	2.67
5.......		2.62	2.56	2.60
1.......	WA-15	2.38	2.35	2.37
2.......		2.62	2.57	2.59
3.......		2.54	2.41	2.48
4.......		2.57	2.51	2.53
5.......		2.52	2.48	2.50

CHROMIUM BY THE DIPHENYLCARBAZIDE (PHOTOMETRIC) METHOD

Laboratory No.	Sample	Chromium, per cent	
		Max	Min
1..........	WA-3	0.16	0.15
2..........		0.165	0.15
3..........		0.18	0.17
4..........		...	...
5..........		0.166	0.155
6..........		0.19	0.16
1..........	WA-22	0.0066	0.0060
2..........		0.02	0.02
3..........		0.005	0.005
4..........		...	...
5..........		0.011	0.008
6..........		0.010	0.010

TABLE II.—RESULTS OF COOPERATIVE TESTS ON THE PROPOSED TENTATIVE METHOD FOR DETERMINATION OF PHOSPHORUS IN HIGH-PHOSPHORUS BRAZING ALLOYS.

Laboratory	Copper, per cent[a]	Silver, per cent[a]	Phosphorus, per cent	
			by Difference	Photometric[b]
A...........	80.07	14.80	5.13	5.03 5.01 5.01
B...........	80.11	14.79	5.10	5.04 5.03 5.04
C...........	80.08	14.79	5.13	5.03 5.04 4.99

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[a] Silver and copper determined in accordance with the Standard Methods for Chemical Analysis of Silver Solders (E 56 – 45).

[b] The average of seven gravimetric determinations of phosphorus gave a value of 5.03 per cent.

TABLE III.—RESULTS OF COOPERATIVE TESTS ON THE PROPOSED TENTATIVE METHODS FOR CHEMICAL ANALYSIS OF COPPER-TELLURIUM ALLOYS.

Laboratory	Copper, per cent	Tellurium, per cent
A................	99.47 99.46 99.47	0.53 0.54 0.54
B................	99.45 99.45 99.48	0.547 0.543
C................	...	0.547 0.547 0.545

TABLE IV.—RESULTS OF COOPERATIVE TESTS ON THE PROPOSED TENTATIVE METHODS FOR CHEMICAL ANALYSIS OF METALLIC MATERIALS FOR ELECTRICAL HEATING.

(All values in per cent.)

Analyst	Sample	Max	Min	Avg	General Average	Certificate Value	Deviation
MANGANESE							
1...	NBS 169	0.078	0.067	0.073	0.0725	0.0730	0.0005
2...		0.072	0.072	0.072			
3...		0.0767	0.0698	0.0718			
4...		0.075	0.071	0.073			
1...	NBS 161	...	...	...	1.289	1.280	0.009
2...		...	...	...			
3...		1.310	1.282	1.300			
4...		1.288	1.256	1.278			
COPPER							
1...	NBS 169	0.015	0.015	0.015	0.0145	0.0150	0.0005
2...		...	...	...			
3...		0.015	0.012	0.014			
4...		...	...	...			
1...	NBS 161	0.044	0.038	0.041	0.041	0.045	0.004
2...		...	...	...			
3...		0.045	0.037	0.041			
4...		...	...	...			

TABLE V.—RESULTS OF COOPERATIVE TESTS ON THE PROPOSED MODIFICATION OF THE BROMATE METHOD FOR ANTIMONY (SECTIONS 15 AND 16) OF METHODS E 57 - 50 T.

Sample (NBS No.)	Amount of Sample Used, g	Antimony, per cent	
		Present	Found
COOPERATOR 1			
127*a*..............	2	0.79	0.79
53*c*..............	0.5	10.19	10.17 10.28
54*c*..............	0.5	7.27	7.22 7.25
COOPERATOR 2			
127..............	2	0.75	0.76 0.77
127*a*..............	2	0.79	0.79 0.79
53*b*..............	1	10.28	10.26 10.32
54*c*..............	1	7.27	7.24 7.29 7.22
COOPERATOR 3			
127*a*..............	2	0.79	0.80 0.78 0.80
53*b*..............	1	10.28	10.24 10.27
54*b*..............	1	7.39	7.40 7.29

TABLE VI.—RESULTS OF COOPERATIVE TESTS ON THE PROPOSED TENTATIVE METHODS FOR CHEMICAL ANALYSIS OF CHROMIUM-COPPER ALLOYS.

Analyst	Sample (Heat No.)	Chromium, per cent			
		Max	Min	Avg	General Average
A.......	53893-5	0.74	0.73	0.733	0.735
B.......		0.740	0.733	0.737	
A.......	53893-6	1.11	1.10	1.11	1.10
B.......		1.10	1.10	1.10	

TABLE VII.—RESULTS OF COOPERATIVE TESTS ON PROPOSED TENTATIVE METHOD FOR DETERMINATION OF LEAD IN ZINC AND ZINC-BASE ALLOYS.

Laboratory	Sample (NBS No.)	Lead, per cent					
		Max	Min	Avg	General Average	Certificate Value	Deviation
C.....	94	0.0326	0.0300	0.031	0.031	0.031	0.000
A.....	94*a*	0.0044	0.0043	0.0043	0.005	0.006	0.001
B.....		0.0066	0.0060	0.0062			
C.....	95	0.324	0.298	0.307	0.31	0.32	0.01
A.....	108	0.048	0.043	0.045	0.045	0.047	0.002
A.....	109	0.0022	0.0021	0.0022	0.0022	0.0020	0.0002

TABLE VIII.—RESULTS OF COOPERATIVE TESTS ON THE PROPOSED TENTATIVE METHOD FOR DETERMINATION OF MAGNESIUM IN ALUMINUM.

(Revision of Methods E 34 - 54 T.)

Sample (NBS No.)	Magnesium, per cent				
	Laboratory A	Laboratory B	Laboratory C	Laboratory D	Certificate Value
85*a*..........	1.56	...	1.57	1.60	1.58
87...........	0.37	0.38	0.38	0.39	0.39

TABLE IX.—RESULTS OF COOPERATIVE TESTS ON THE PROPOSED TENTATIVE METHOD FOR DETERMINATION OF ALUMINUM IN ELECTRONIC NICKEL.

(Revision of Methods E 107 - 54 T.)

Sample (Melt No.)	Aluminum, per cent				
	Laboratory A	Laboratory B	Laboratory C	Laboratory D	Laboratory E
202..........	0.009	0.010	0.011	0.011	0.013
1025.........	0.092	0.092	0.092	0.095	0.090
8153.........	0.002	0.001	0.003	0.002	0.003

TABLE X.—RESULTS OF COOPERATIVE TESTS ON THE PROPOSED TENTATIVE METHOD FOR DETERMINATION OF COLUMBIUM AND TANTALUM IN STAINLESS STEEL.

(Revision of Methods E 30 – 54 T.)

	NBS Sample No. 123*b*			Special Sample		
	Columbium, per cent	Tantalum, per cent	Tungsten, per cent	Columbium, per cent	Tantalum, per cent	Tungsten, per cent
Certified value	0.75	0.20	0.18	....	....	....
Laboratory						
1	0.759	0.205	0.17	0.790	0.224	—0.01
	0.754	0.203	0.16	0.785	0.216	—0.01
	0.769	0.205	0.18	0.766	0.220	—0.01
2	0.722	0.208	...	0.728	0.220	...
	0.712	0.204	...	0.728	0.228	...
	0.716	0.210	...	0.720	0.228	...
	0.726	0.210	...	0.734	0.230	...
3	0.76	0.21	0.15	0.77	0.28	none
	0.71	0.20	0.16	0.79	0.26	none
	0.76	0.22	0.16	0.77	0.27	none
4	0.72	0.22	...	...	...	...
	0.73	0.22	...	...	...	...
	0.73	0.22	...	...	...	...
	0.74	0.21	...	...	...	...
	0.76	0.21	...	...	...	...
	0.76	0.21	...	...	...	...

TABLE XI.—RESULTS OF COOPERATIVE TESTS ON THE PROPOSED REVISION OF THE PHOSPHATE METHOD FOR MAGNESIUM (SECTION 16) IN METHODS E 47 – 45.

Sample	Magnesium Present, per cent	Magnesium Found, per cent		
		Cooperator 1	Cooperator 2	Cooperator 3
NBS 94*a*	0.042	0.042	0.049	0.040
		0.042	0.049	0.042
		0.042	0.049	0.042
				0.042
				0.043
				0.043
5 g of NBS 94*a* + 5 g of NBS 109	0.021	...	0.025	0.022
		...	0.026	0.022
		...	0.026	
10 g of NBS 94*a* + 0.25 g of copper	0.042	...	0.049	0.042
		...	0.049	0.042
		...	0.049	
	Magnesium Present, g	Magnesium Found, g		
Synthetic alloy of 9.6 g zinc + 0.4 g of aluminum plus magnesium	0.00300	0.00302	...	...
	0.00300	0.00293	...	...
	0.00600	0.00580	...	...
	0.00600	0.00596	...	...

TABLE XII.—RESULTS OF COOPERATIVE TESTS ON THE PROPOSED TENTATIVE METHOD FOR DETERMINATION OF MAGNESIUM IN CAST IRON.

(Revision of Methods E 30 – 54 T.)

Laboratory No.	Sample	Magnesium, per cent		
		Max	Min	Avg
1	1[a]	0.040	0.036	0.038
2		0.042	0.036	0.039
1	2[b]	0.083	0.068	0.075
2		0.084	0.071	0.077
1	3[c]	0.075	0.059	0.068
3		0.069	0.062	0.065

[a] *On Sample No. 1*, spectrographic values of 0.038, 0.036, and 0.037 per cent were obtained by Laboratory No. 4, and 0.037 per cent spectrographically and 0.037, 0.037 per cent using the magnesium pyrophosphate method by Laboratory No. 2.

[b] *On Sample No. 2*, spectrographic values of 0.083, 0.086, and 0.089 per cent were reported by Laboratory No. 4. On this sample Laboratory No. 2 obtained 0.075, 0.075, and 0.075 per cent by the magnesium pyrophosphate method.

[c] *On Sample No. 3*, a spectrographic value of 0.069 per cent was reported by Laboratory No. 3.

TABLE XIII.—RESULTS OF COOPERATIVE TESTS ON THE PROPOSED TENTATIVE HYDROBROMIC ACID METHOD FOR COPPER IN ZINC-BASE DIE-CASTING ALLOYS.

(Revision of Methods E 47 – 45.)

Sample	Sample Weight, g	Copper, per cent		
		Cooperator 1	Cooperator 2	Cooperator 3
NBS sample 108 (Certificate value, Cu = 0.0004 per cent)	10	...	...	0.000449
	10			0.000477
	10			0.000487
NBS sample 109 (Certificate value, Cu = 0.0005 per cent)	5	0.00060	...	...
	10	0.00050	...	0.000487
	10	0.00055		0.000543
NBS sample 110 (Certificate value, Cu = 0.0031 per cent)	5	0.0031	...	...
	10	...	...	0.00335
	10			0.00339
	10			0.00342
	10			0.00361
NBS sample 94*a* (Certificate value, Cu = 1.08 per cent)	0.050	1.07	...	...
	0.100	1.08	...	...
	0.250	1.08	...	...
	0.5/10	...	1.08	...
	0.5/10		1.09	
	0.5/10		1.09	
1 g NBS sample 109 + 0.006 g NBS sample 94*a*, Cu = 0.0070 per cent		...	0.0075	...
1 g NBS sample 109 + 0.0055 g NBS sample 94*a*, Cu = 0.0065 per cent		...	0.0070	...
1 g NBS sample 109 + 0.0058 g NBS sample 94*a*, Cu = 0.0068 per cent		...	0.0072	...

REPORT OF COMMITTEE E-4
ON
METALLOGRAPHY*

Committee E-4 on Metallography held two meetings during the year: on June 29, 1955, during the Annual Meeting of the Society, and on February 28, 1956, during ASTM Committee Week.

A number of changes in membership have occurred, and the committee now consists of 112 members.

The officers elected for the ensuing term of two years are as follows:

Chairman, L. L. Wyman.
Vice-Chairman, R. E. Penrod.
Secretary, Mary R. Norton.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1955 Annual Meeting, Committee E-4 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Method for:

Estimating the Average Grain Size of Metals (E 112 - 55 T), and

Revision and Reversion to Tentative of:

Standard Definitions of Terms Relating to Metallography (E 7 - 27).

These recommendations were accepted by the Standards Committee on September 15, 1955, and the new method and revised definitions appear in the 1955 Book of ASTM Standards, Part 2.

The preparation of Method E 112 is the result of a sustained effort which was initiated in 1937. Included in the revised Definitions E 7 are nearly a thousand terms used in the field of metallography.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Selection and Preparation of Samples (F. C. Hull, chairman) has been working on revisions of practices for the selection and preparation of samples, and nearly completed is the section on electrolytic polishing and etching.

Because of the need to greatly strengthen the recommended procedures for macro-etch testing, a special section has been set up for this work. In addition, this group will follow up the current survey on the desirability of macro-etch standards, at the request of the Administrative Committee on Simulated Service Testing.

Subcommittee III on Nomenclature (P. A. Beck, chairman) has prepared what appears to be an acceptable means of designating the phases in alloy systems, and the document will be discussed with experts in England prior to being presented to the main committee.

Subcommittee IV on Photomicrography (S. W. Poole, chairman) is preparing for the 1956 Photomicrographic Exhibit. The great interest and success of the student competition participation, as well as the excellent quality of the exhibits submitted, make this the country's outstanding display of quality photomicrographs.

Subcommittee V on Microhardness (W. E. Shebest, chairman) is evaluating its last round-robin tests on standard test-plate samples and is continuing a comparable series of tests with specially calibrated micrometers and test plates.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

Subcommittee VI on X-ray Diffraction (W. L. Fink, chairman).—The many advances in both technique and equipment in recent years have necessitated an extensive enlargement of coverage of the Recommended Practice for Identifying of Crystalline Materials by the Hanawalt X-ray Diffraction Method (E 43 - 49 T), and in connection with this work a group of diffraction experts have volunteered their services to the committee. This situation resulted from the close cooperation between Committee E-4 and the Joint Committee on Chemical Analysis by Powder Diffraction Methods, sponsors of the well-known Card File Index.

Subcommittee VII on Thermal Analysis (D. I. Finch, chairman) is in the initial stages of preparing a comprehensive recommendation on resistance methods of analysis.

Subcommittee VIII on Grain Size (R. E. Penrod, chairman) has completed the major task of preparing the new Method E 112 referred to previously in this report.

It is now working on a series of reference photographs for small amounts of disperse phases and similar appearing voids or particles, possibly including minute inclusions such as in vacuum melted metals, porosity, etc.

Subcommittee X on Decarburization (J. J. B. Rutherford, chairman) which was organized particularly to cooperate with similar interests in other technical societies, reports a complete dearth of such activities, and therefore it has been disbanded.

Subcommittee XI on Electron Microstructure (R. M. Fisher, chairman) is continuing a most active program on the studies of techniques and microstructures of steel and on the structures of non-ferrous materials.

In conjunction with the 1956 Annual Meeting, the subcommittee will sponsor a technical session relating to its current efforts.

This report has been submitted to letter ballot of the committee, which consists of 112 members; 79 members returned their ballots, of whom 75 have voted affirmatively and 2 negatively.

Respectfully submitted on behalf of the committee,

L. L. WYMAN,
Chairman.

MARY R. NORTON,
Secretary.

APPENDIX I

ELECTRON MICROSTRUCTURE OF SOME QUENCHED-AND-TEMPERED, LOW-ALLOY, MEDIUM-CARBON STEELS*†

By R. P. Sernka[1] and S. T. Ross[2]

In connection with other work done at the Chrysler Engineering Research Laboratories (**11**),[3] there has been the opportunity to investigate the electron microstructure of six low-alloy medium-carbon steels. This phase of the work was undertaken so that microstructure could be correlated with data obtained from physical tests. It was also felt that information about the products formed during the tempering of martensite in various alloy steels was incomplete. The specimens (SAE 2340, 4052, 4063, 4140, 4340 and 5150 steels) were quenched and then tempered for various times. Steel analyses and the heat treatments for each specimen are shown in Tables I and II.

Specimen Preparation

All specimens were copper plated 0.0005 in. to prevent decarburization during heat treatment. The specimens were torsion test bars as described in reference (**11**). Heat treatment was accomplished in a muffle furnace provided with a cracked-gas atmosphere. The air-gas ratio was held at 3:1 at the cracker so that as nearly neutral a furnace atmosphere as possible was obtained. All specimens were austenitized for 1 hr and quenched in agitated oil. Tempering was done in a pot-type circulating air furnace, and the bars were air cooled after removal.

Electron Micrographic Technique

Examination was made of cross-sections cut from cylindrical areas between the shanks of torsion bars. The bars were submerged in the coolant while the cutting was performed by an abrasive wheel. To prevent further structural change, those bars tempered in the 200 to 350 F range were not mounted in bakelite but were clamped mechanically. Likewise, polishing of these specimens was done with care, using coolants to prevent surfaces from rising above original tempering temperatures.

Formvar replicas were taken after surfaces had been etched with a 4 per cent picral solution containing 0.1 per cent zephiran chloride. The replicas were shadowed with chromium at an angle of approximately 45 deg. Figures 1 to 8

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* Presented at a meeting of Subcommittee XI an Electron Metallography of ASTM Committee E-4 on Metallography, Atlantic City, N. J., June 19, 1956.

† This paper is taken from a thesis submitted by R. P. Sernka in partial fulfillment of the requirements for the degree of M.S. in Metallurgy at Wayne University.

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[2] Supervisor, Metallurgical Research, Chrysler Corp., Detroit, Mich.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 408.

TABLE I.—CHEMICAL ANALYSES OF STEEL SAMPLES, PER CENT.

Steel	Carbon	Manganese	Phosphorus	Sulfur	Silicon	Chromium	Nickel	Molybdenum
SAE 2340	0.40	0.83	0.015	0.030	0.25	0.05	3.50	. . .
SAE 4052	0.50	0.79	0.015	0.020	0.25	0.05	. . .	0.25
SAE 4063	0.65	0.86	0.015	0.018	0.25	. . .	. . .	0.24
SAE 4140	0.42	0.92	0.020	0.026	0.26	0.98	. . .	0.19
SAE 4340	0.40	0.73	0.010	0.020	0.35	0.88	1.68	0.25
SAE 5150	0.51	0.77	0.015	0.020	0.20	0.78	. . .	0.20

TABLE II.—SPECIMEN HEAT TREATMENT.

Steel	Specimen Number	Austenitizing Temperature, deg Fahr	Tempering Temperature, deg Fahr	Tempering Time, hr	Avg. Rockwell Hardness, C scale
SAE 2340	106 to 108	1450	950	1	38.0
SAE 2340	109 to 111	1450	825	1	41.0
SAE 2340	112 to 114	1450	720	1	43.0
SAE 2340	115 to 117	1450	600	1	48.0
SAE 2340	118 to 120	1450	400	1	54.0
SAE 2340	121 to 123	1450	200	1½	60.0
SAE 2340	124 to 126	1450	As Quenched	. . .	62.0
SAE 4052	22 to 24	1500	1040	1	36.5
SAE 4052	25 to 27	1500	915	1	41.0
SAE 4052	28 to 30	1500	765	1	47.0
SAE 4052	31 to 33	1500	620	1	52.0
SAE 4052	34 to 36	1500	450	1	56.0
SAE 4052	37 to 39	1500	200	1½	63.0
SAE 4052	40 to 42	1500	As Quenched	. . .	64.5
SAE 4063	13 to 15	1450	1080	1½	36.0
SAE 4063	10 to 12	1450	970	1½	40.5
SAE 4063	7 to 9	1450	830	1½	45.0
SAE 4063	4 to 6	1450	560	2	52.5
SAE 4063	1 to 3	1450	440	2	57.5
SAE 4063	16 to 18	1450	350	1	61.0
SAE 4063	19 to 21	1450	As Quenched	. . .	65.0
SAE 4140	64 to 66	1550	1075	1	35.5
SAE 4140	67 to 69	1550	950	1	40.5
SAE 4140	70 to 72	1550	800	1	46.0
SAE 4140	73 to 75	1550	600	1	50.0
SAE 4140	76 to 78	1550	450	1	53.0
SAE 4140	79 to 81	1550	250	1½	60.0
SAE 4140	82 to 84	1550	As Quenched	. . .	61.0
SAE 4340	85 to 87	1550	1075	1	36.0
SAE 4340	88 to 90	1550	950	1	40.5
SAE 4340	91 to 93	1550	800	1	44.5
SAE 4340	94 to 96	1550	600	1	49.0
SAE 4340	97 to 99	1550	450	1	52.0
SAE 4340	100 to 102	1550	250	1½	58.0
SAE 4340	103 to 105	1550	As Quenched	. . .	59.0
SAE 5150	43 to 45	1500	1175	1	29.0
SAE 5150	46 to 48	1500	1060	1	33.0
SAE 5150	49 to 51	1500	925	1	39.0
SAE 5150	52 to 54	1500	720	1	47.5
SAE 5150	55 to 57	1500	500	1	53.0
SAE 5150	58 to 60	1500	300	1½	60.5
SAE 5150	61 to 63	1500	As Quenched	. . .	61.0

are electron micrographs of these specimens at magnifications of 30,000 diameters.

ELECTRON MICROSTRUCTURES

Structures representative of those specimens tempered at 200 to 350 F are illustrated in Figs. 1 to 3. The tempered martensite needles contain many fine carbide particles and are outlined by films. The nature of these particles has been indicated by the work of Jack (1), Hagg (2) Austin and Schwartz (3), and others. There is doubt as to their exact composition or if more than one form is found during tempering. Therefore, in this paper the generic term percarbides has been used. Lighter-etching areas around the percarbides are regarded as regions of lower carbon solid solution as suggested by Kurdjumov and Lyssak (4). Films around the needles are also considered to be composed of percarbide, since, according to data presented by

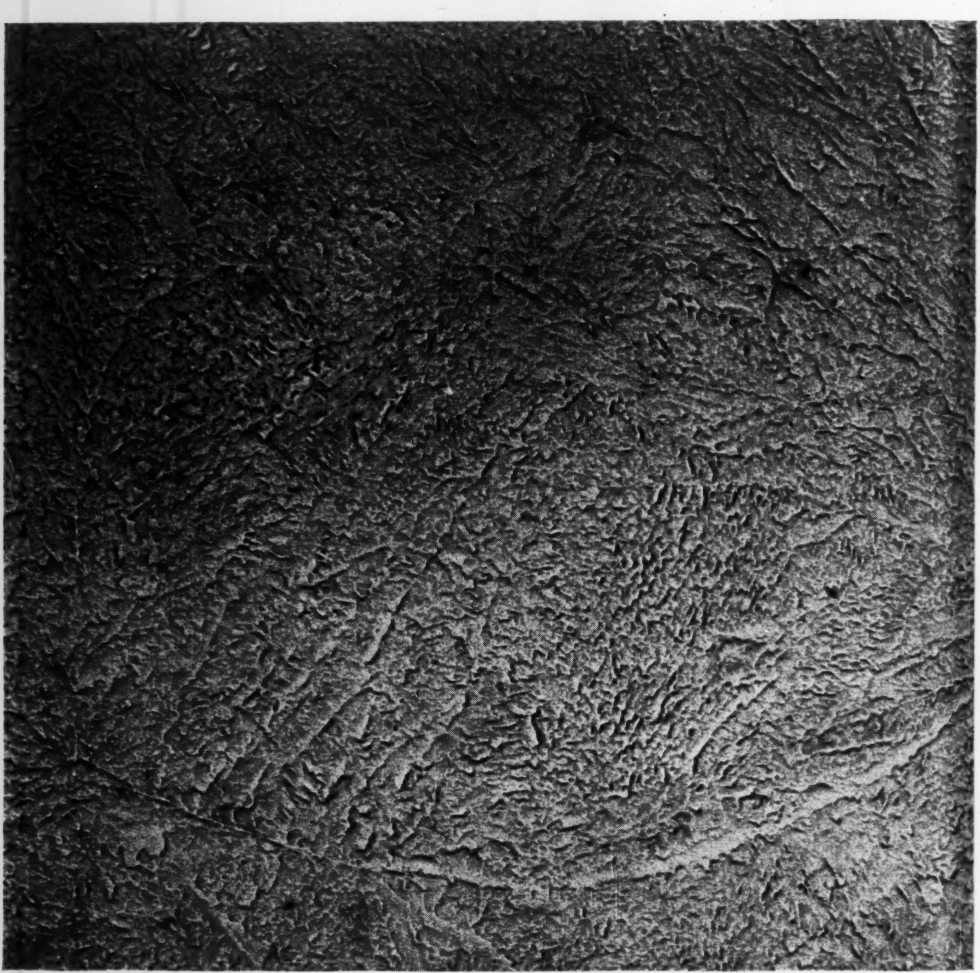

FIG. 1.—SAE 4063 Steel, Tempered at 350 F for 60 min (×30 000).

Lement, Averbach and Cohen (5), cementite is not present in steels tempered at such low temperatures. Needles partially outlined by percarbide films are most clearly seen in Fig. 2. The matrix material is considered to be ferrite supersaturated with carbon as indicated by Kurdjumov and Lyssak (6). No indications of retained austenite were found in any of the samples examined.

Figure 4 is an electron micrograph of SAE 5150 steel tempered at 300 F and purposely overetched before replication. Comparison with Fig. 3 of the same specimen, properly etched, indicates a very apparent difference in structure. It will be noted that when the specimen is etched too deeply, fine percarbides no longer appear as discrete particles. Instead, an artefact network is produced.

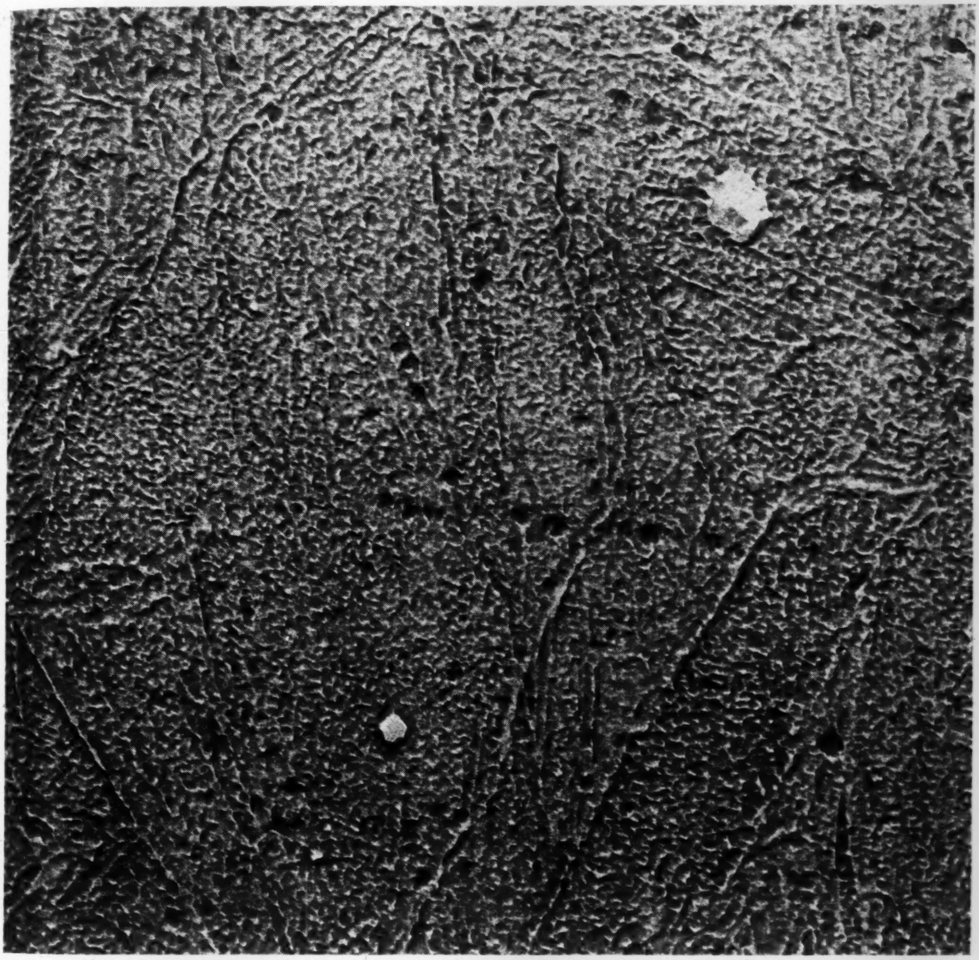

FIG. 2.—SAE 4140 Steel, Tempered at 250 F for 90 min (×30 000).

Typical electron micrographs of those specimens tempered in the 400 to 500 F range are found in Figs. 5 and 6. Examination indicates that both contain two sizes of carbide particles. The fine percarbides appear to have been partially supplanted by coarser particles of cementite. Also, percarbide films which outlined the martensite needles at lower tempering temperatures have partially changed to large, elongated, non-continuous cementite particles.

FIG. 3.—SAE 5150 Steel, Tempered at 300 F for 90 min (×30 000).

Figures 7 and 8 are electron micrographs representative of specimens tempered at 600 F and higher. Cementite particles are seen in a matrix of smooth ferrite. Very few percarbide particles are noted. No significant differences are detected among structures of the various steels tempered under the same conditions.

DISCUSSION

Muir (7) noted that instability in elastic limit-hardness data coincided with the formation of cementite films in the 450 to 500 F tempering range as reported by Lement (5). Muir also regarded improvement in his data for samples tempered above 500 F as a function of the relief of quenching and transformation stresses. Nevertheless, the present investigation has shown that such cementite films are not formed during the tempering process. Figures 1 to 3 indicate that percarbide films are present at tempering temperatures as low as 250 F. However, Figures 5 and 6 show that these films are gradually supplanted by large, elongated, but discontinuous cementite particles at temperatures above 450 F after one hour or more of tempering.

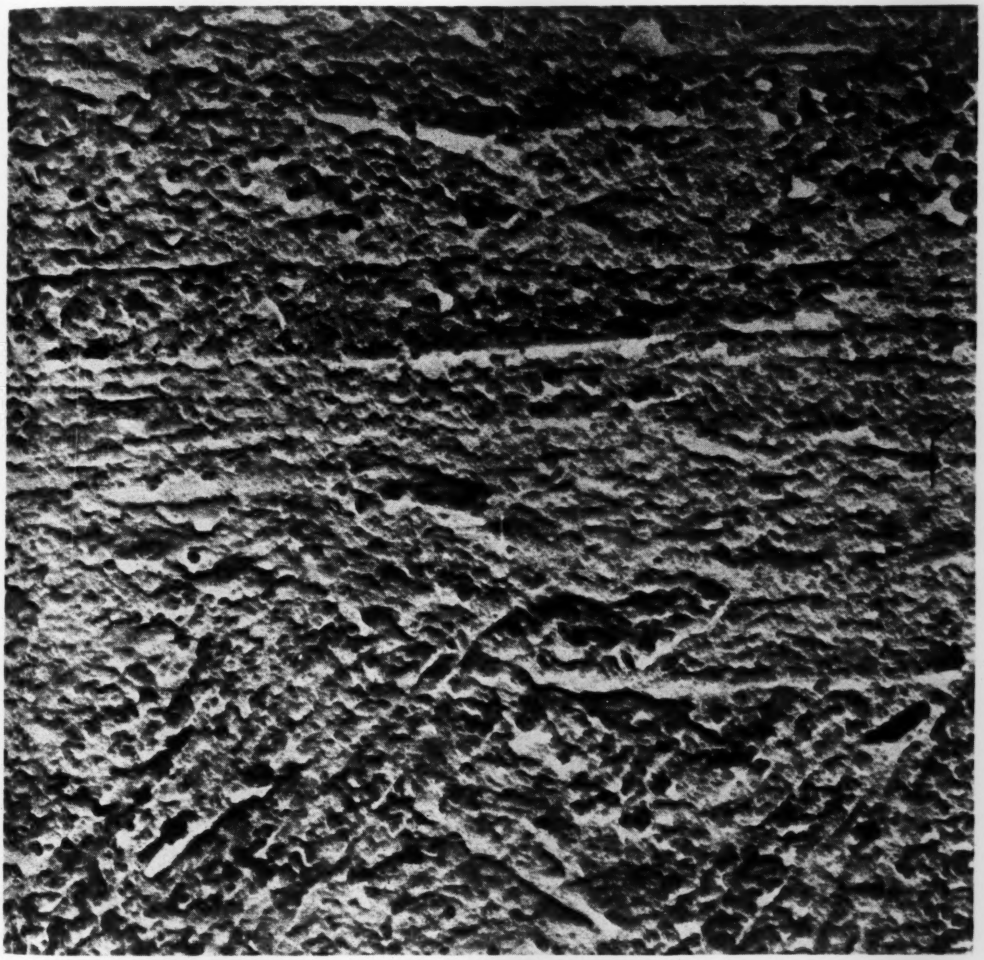

FIG. 4.—SAE 5150 Steel, Tempered at 300 F for 90 min (×30 000).

Identification of the films outlining martensite needles at low tempering temperatures agrees with the view of Kurdjumov and Lyssak (4), namely, that formation of percarbides can begin at martensite boundaries in the 200 to 300 F tempering range.

It has been pointed out by Ross in his discussion of the Lement paper (8) that and supersaturated ferrite are intermediate products in the decomposition of martensite to ferrite and cementite during the tempering process. However, the concept of three distinct stages in tempering has been based on temperature only. When both temperature and time dependence are considered, the limits dividing tempering must be very broad, if existent at all. The authors agree with the concept that percarbides Figs. 1 to 3 indicate that percarbides do not form a continuous subgrain network in martensite. This is not in agreement with the consideration by Lement *et al*, that the appearance and subsequent dissolution of such a network is a factor in the process of tempering.

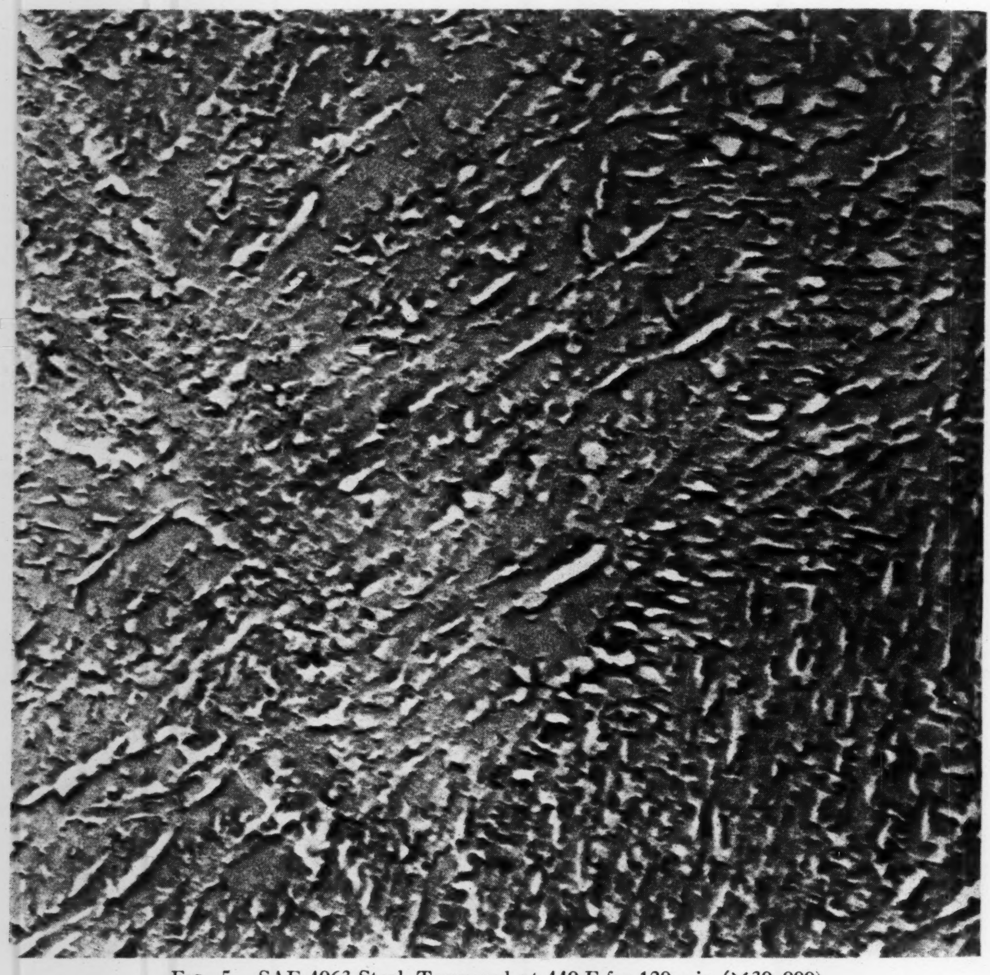

FIG. 5.—SAE 4063 Steel, Tempered at 440 F for 120 min (×30 000).

In a previous paper (9), the authors have referred to fine carbides in martensite associated with bainite in end-quench bars as being indicative of a subgrain structure. Refinements in metallographic techniques have resulted in electron micrographs which indicate that subgrains do not exist in tempered 100 per cent martensite nor in partially transformed areas of hardenability bars. Effects caused by improper surface preparation and regions of lower carbon solid solution have led to identification of an artifically produced network and its incorrect representation as a martensite subgrain structure.

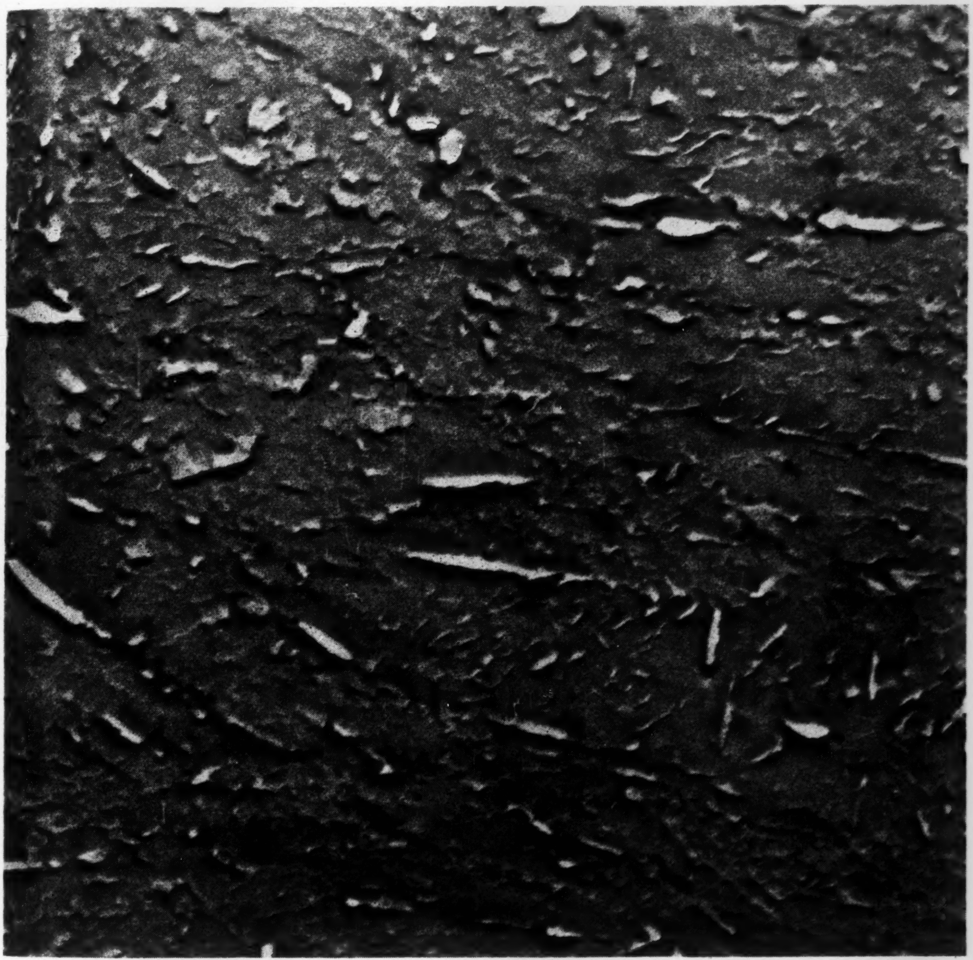

FIG. 6.—SAE 4340 Steel, Tempered at 450 F for 60 min (×30 000).

The importance of sample preparation in electron metallography cannot be emphasized too highly. The extremely fine-tempered martensite structures will not be revealed properly unless great care is taken that every trace of disturbed metal is removed by etching. Further, to prevent misrepresentation of the actual structure, the etch must be only of sufficient depth to delineate the particles. As reported by Subcommittee XI of ASTM Committee E-4 (10) and as is indicated in Figs. 3 and 4, shadowing effects also will mask the true structure if overetching has occurred.

It is of significance that the steels studied in this investigation, when tempered at the same temperature and for the same length of time, display the same microstructure. It appears that, while the amount of carbide particles in tempered martensite is determined by chemical composition, their size and distribution is a function of the time and temperature of tempering.

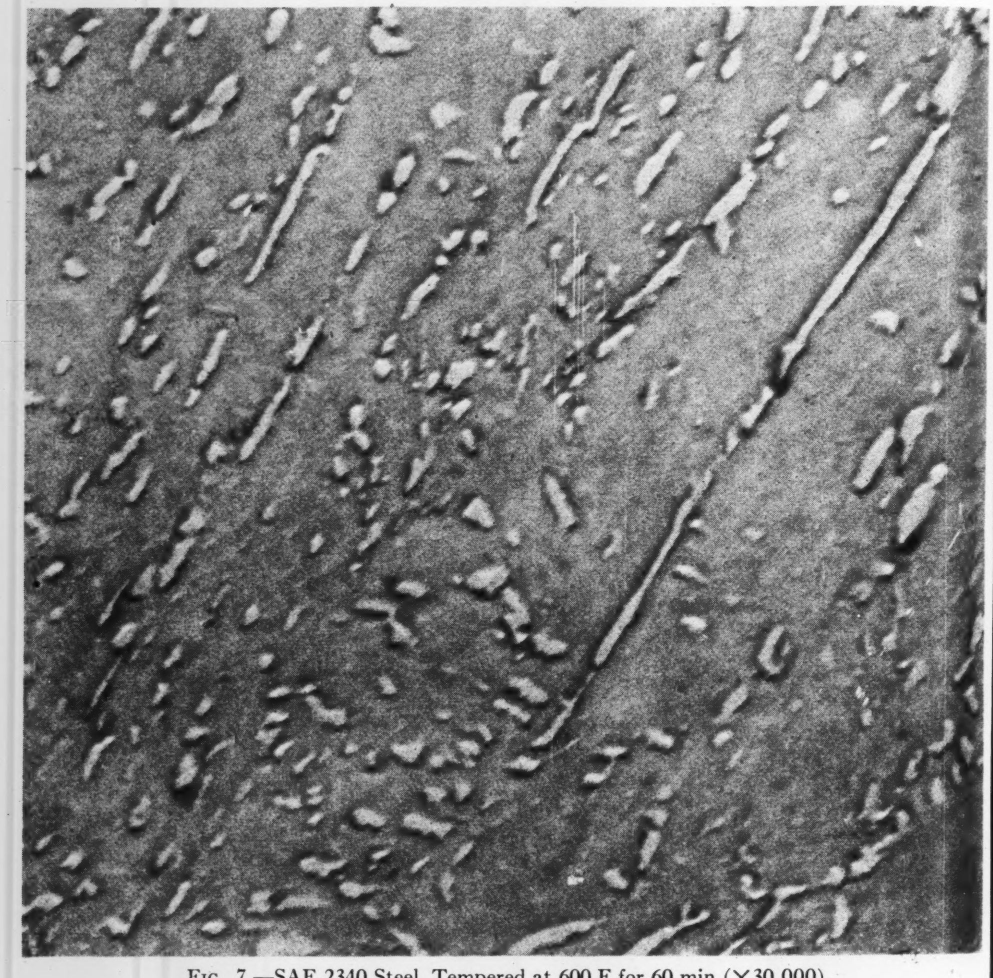

FIG. 7.—SAE 2340 Steel, Tempered at 600 F for 60 min (×30 000).

CONCLUSIONS

Consideration of the results of this investigation has led to the following conclusions:

1. Electron micrographs show that percarbide films outline martensite needles tempered for 1 hr or more in the 200 to 350 F range. Above 450 F, these films are supplanted by elongated and discontinuous cementite particles.

2. No martensite subgrain network was found in electron micrographs of the samples.

3. No microstructural differences were detected in electron micrographs of various steels tempered under the same conditions of time and temperature.

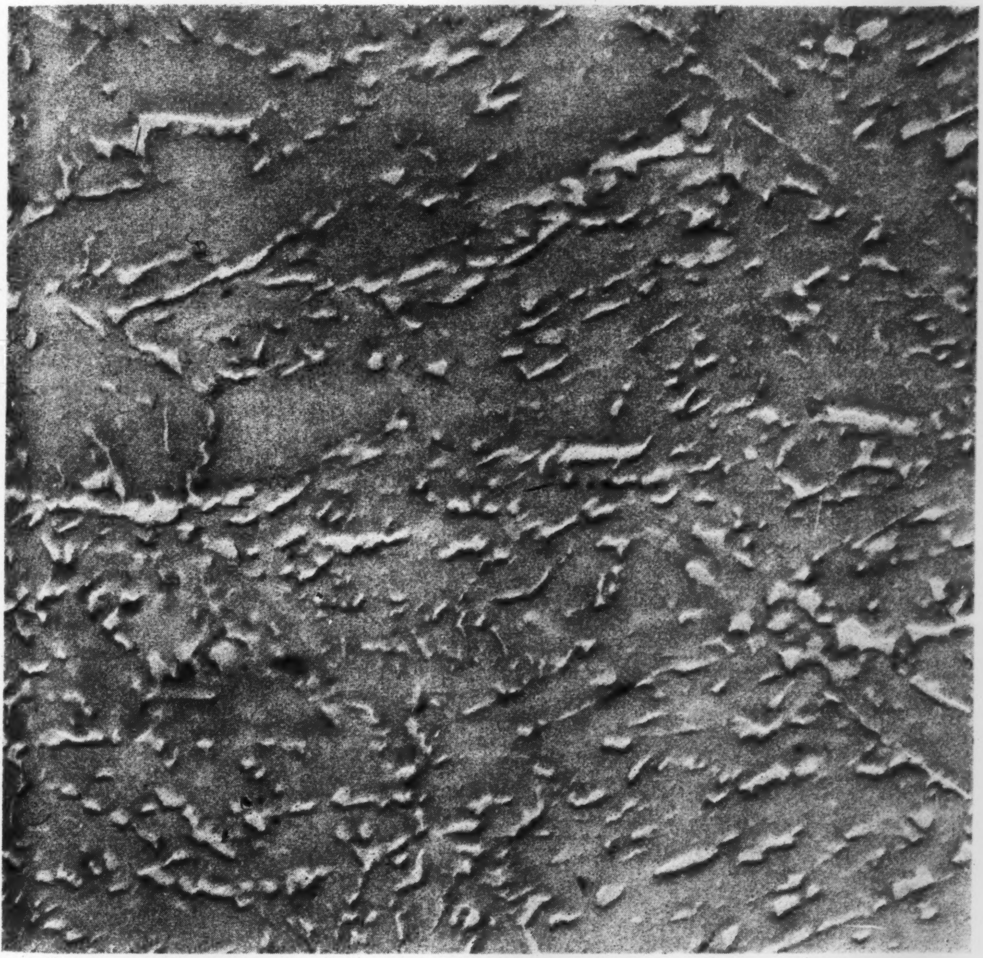

FIG. 8.—SAE 4063 Steel, Tempered at 830 F for 90 min (×30 000).

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APPENDIX II

A METHOD FOR MAKING POSITIVE REPLICAS AND THE PREPARATION OF STEEL SPECIMEN FOR ELECTRON MICROSCOPY*

By J. B. Le Poole[1] and F. van Wijk[2]

The method described below was used for the investigation of steel samples furnished by Subcommittee XI on Electron Microstructure of Metals of ASTM Committee E 4. The method was developed by Dalitz, Schuchmann and Beekhuis (**1**).[3] The two main reasons for using a positive replica are: (1) the great ease with which the first silver replica is stripped from the etched specimen and (2) the fact that a positive replica must necessarily give a more real picture. The preparation of the specimens before etching was done in a conventional way.

Preparation and Etching

The rather thin specimens were mounted on bakelite with the aid of perspex. After grinding on the finest paper, polishing was initially accomplished by means of chromium oxide, followed by alumina, alternated by nital etchings. The etching was generally done in a solution of 4 per cent picric acid in ethyl alcohol containing a small quantity of zephiran chloride. It was found that very frequently the results of etching were improved when the picric acid and the alcohol were thoroughly dried beforehand. The use of zephiran chloride made the specimens much cleaner. After etching, the specimens were washed and dried according to *Sequence A* given in the Appendix to the First Progress Report of Subcommittee XI.[4]

In a few cases the etching was not quite satisfactory, and improvement was obtained by adding some more zephiran chloride or a little water, and in exceptional cases, etching was done in 2 per cent nital with zephiran chloride. In the last instance pure dry ethyl alcohol was used as well for making the etching solution, and the specimen was cleaned by stripping a collodion film from it at least twice.

The Negative Silver Replica

The technique of evaporating silver on to the specimen in a vacuum of 10^{-4} mm mercury is shown schematically in Fig. 1. The silver is melted in a small conical crucible. This crucible is made by winding a tungsten wire to shape, bright annealing the wire in a vacuum of less than 10^{-2} mm mercury, and then coating it with alundum cement.[5] The alundum is mixed with acetone to form a paste which is applied with a brush to the coiled wire until a nice smooth crucible is formed. The crucible is heated several times in a vacuum of 10^{-4} mm mercury to make it resistant to temperature up to 2900 F (1600 C). For regular use in evaporating silver at about 1830 F (1000 C), a useful life of 4 to 6 months is normal for these crucibles. It takes about 2 hr to manufacture such a crucible.

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* Presented at a meeting of Subcommittee XI on Electron Microstructure of ASTM Committee E-4 on Metallography, Atlantic City, N. J., June 19, 1956.

[1] E. M. Div., Technical Physics Dept., T.N.O.-T.H., Delft, Holland.

[2] Metaalinstituut, T.N.O., Delft, Holland.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 414.

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 50, p. 490 (1950).

[5] The Morgan Cucible Co., Ltd.

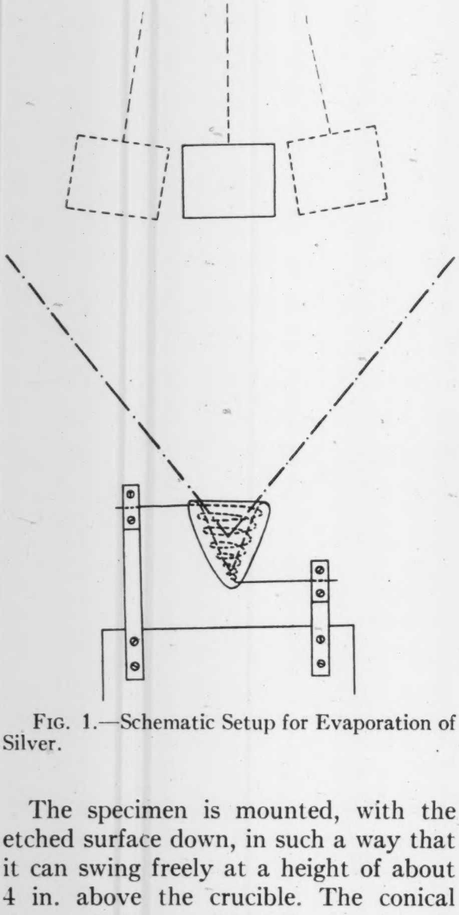

FIG. 1.—Schematic Setup for Evaporation of Silver.

The specimen is mounted, with the etched surface down, in such a way that it can swing freely at a height of about 4 in. above the crucible. The conical shape of the crucible helps to concentrate the rays of silver atoms in the direction of the specimen. Regular rocking of the specimen results in an evenly distributed layer of silver that follows exactly all irregularities of the specimen. If the heat is not carried off quickly enough from the specimen, it is necessary to apply the silver layer at intervals to prevent the development of a structure in the silver (2). Generally in two periods of 3 min each a silver replica of about 7 μ thickness was obtained. Figure 2 gives a picture of the setup under the vacuum jar.

THE POSITIVE CARBON REPLICA

After the specimen has been taken out of the vacuum jar, the silver replica is easily stripped and mounted on a glass plate with the aid of some small pieces of cellotape. Formerly, the carbon replica was obtained by a glow discharge in xylene vapor, but for the past year Bradley's method (3) has been used. In this two carbon electrodes of 0.2 in. diameter, one with a sharp point and one flat, are pressed together by a spring and the silver replica is mounted vertically. The setup under the vacuum jar is shown in Fig. 3. Several pulses of a strong electric current blow off an amount of carbon which can be estimated as fol-

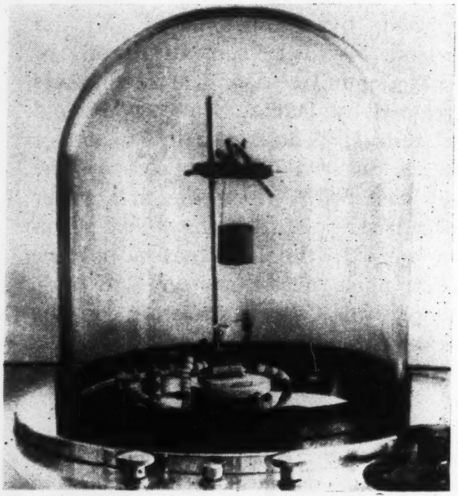

FIG. 2.—Evaporation of Silver for First Replica.

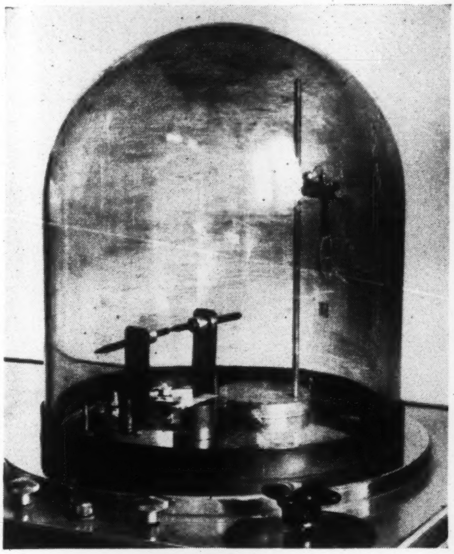

FIG. 3.—Evaporation of Carbon for Second Replica.

lows. A flat piece of white porcelain or opal glass on which a drop of high vacuum oil (silicon oil) is applied serves as an indicator. Since the carbon does not settle on the oildrop, a contrast can be seen as soon as the carbon layer on the porcelain reaches a thickness of about 100 Å.

The replica is removed from the vacuum jar when enough carbon has been deposited, and is then cut into pieces of the size of a Kodak copper grid. The pieces are placed, carbon side up, on the surface of a solution consisting of 40 parts by volume of concentrated nitric acid in 60 parts by volume of distilled water. To make sure that the silver is completely dissolved, the carbon replica not disturbed by surface stresses, and to clean the replica from all traces of nitric acid solution, the following sequence of baths is used:

10 min in 40 vol per cent nitric acid
10 min in concentrated nitric acid
some seconds in 30 vol per cent nitric acid

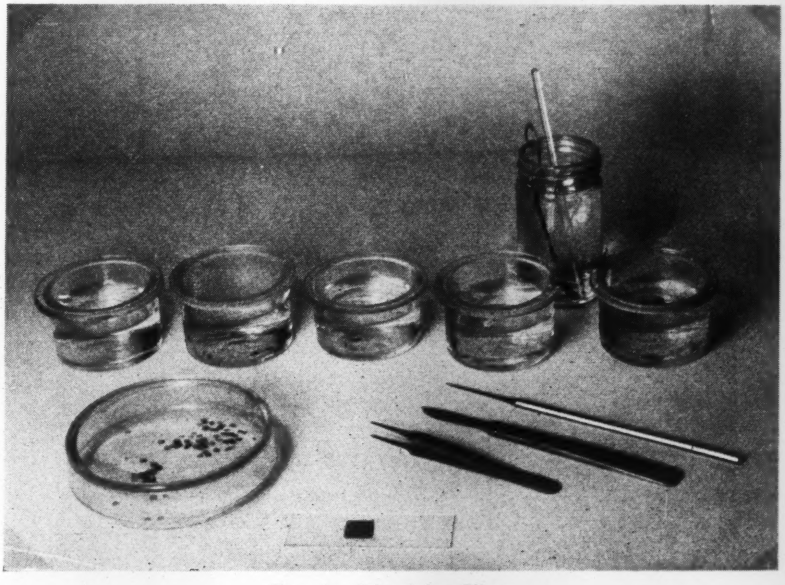

FIG. 4.—Dissolving Silver.

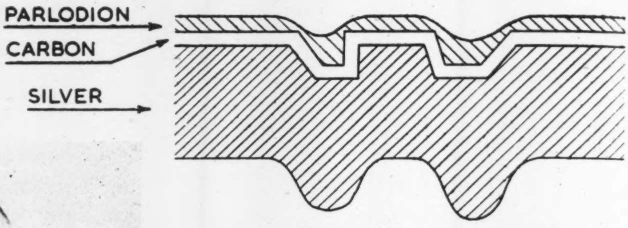
PARLODION
CARBON
SILVER

FIG. 6.—Parlodion Backing of Carbon Replica

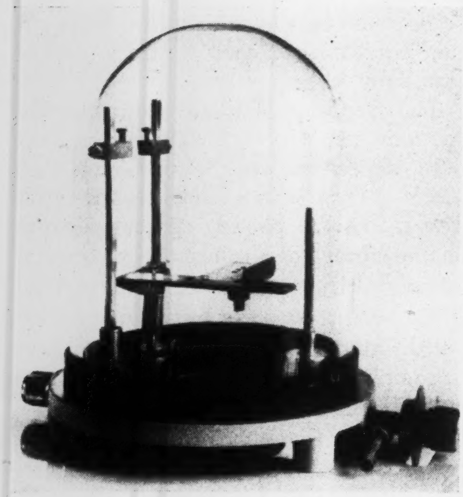

FIG. 5.—Shadow-casting.

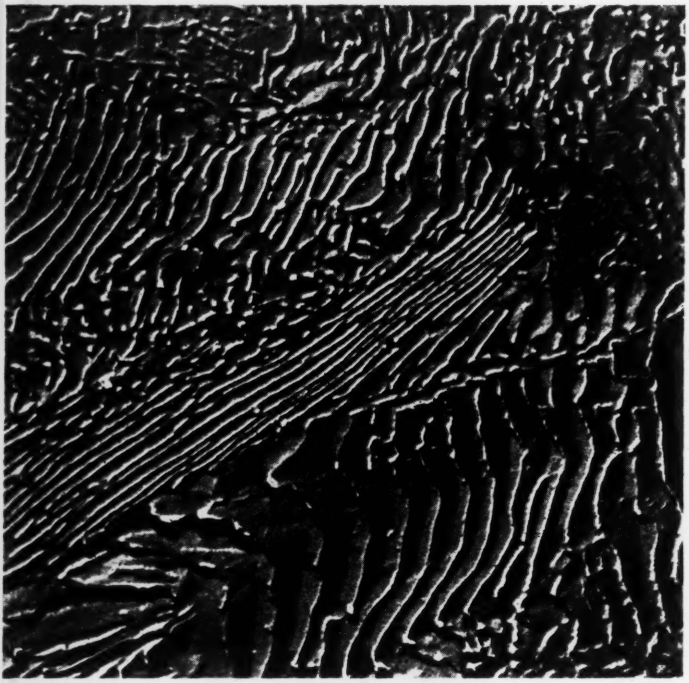

FIG. 7.—Cold-Drawn Patented Steel Wire (× 8000).

Note: Close-up after being reduced one-half.

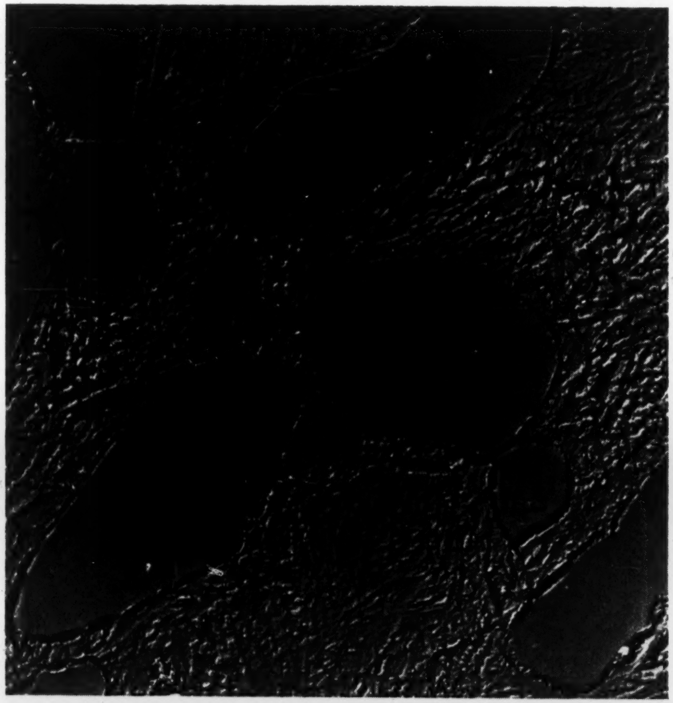

FIG. 8.—High Carbon High Chromium Tool Steel in the Hardened Condition (× 8000).
Note: Close-up after being reduced one-half.

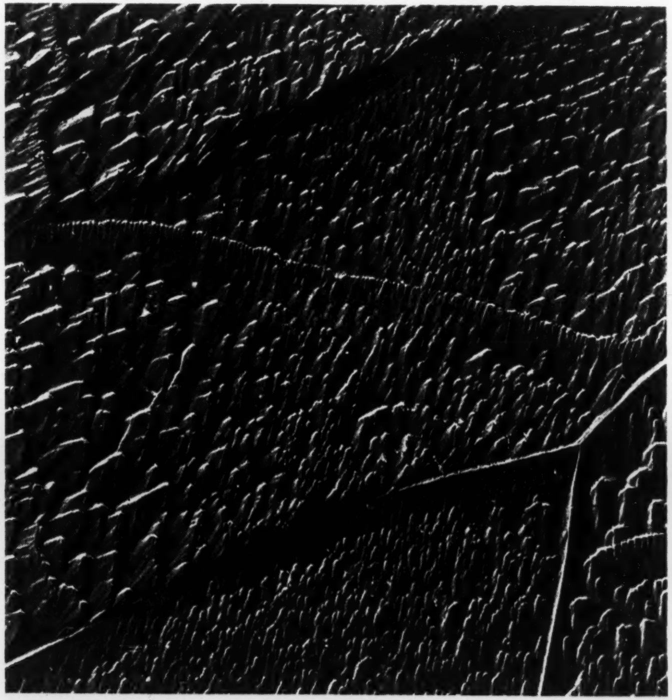

FIG. 9.—Subboundaries and Slip-Lines in Mild Steel 6 per cent stressed After Etchings.
(× 8000).
Note: Close-up after being reduced one-half.

10 min in distilled water
5 min in distilled water

Figure 4 shows the baths and equipment. It is evident that the nitric acid and distilled water used should be free of Cl^- ions. After washing in distilled water, the carbon replicas are mounted on Kodak supporting grids.[6]

SHADOW-CASTING

The positive carbon replicas, mounted on a supporting grid, are shadowed with tungsten oxide in the following manner. A tungsten wire is bright annealed in vacuum and then heated several times for about 10 sec in air to a dull red. After cooling, the degree of oxidation can be judged by the color of the wire (a citron yellow color is preferred). Having obtained this color, the amount of oxide on the wire is such that, if evaporated completely, it produces a convenient degree of shadowing. Generally the setup in vacuum, as shown in Fig. 5, is chosen so as to shadow the specimens under an angle of 45 deg.

CONCLUSIONS AND EXAMPLES

The total time between final etching and replicas ready for use in the electron microscope is about $2\frac{1}{2}$ hr. The method described above does not take much more time than does the preparation of shadowed negative replicas. An important advantage of using a final carbon replica is that it has a good resistance against heating by the electron beam, as does the tungsten oxide used for shadow-casting. If the carbon replica is not very thin the contrast may, at places with steep slopes, be even too great and an effect known as "snow-caps" results. In such a case improvement may be obtained by a method given by Beekhuis consisting of backing the carbon replica with a layer of parlodion in the manner schematically shown in Fig. 6. An electron micrograph resulting from a replica prepared in that way is shown in Fig. 7. It shows the microstructure of cold-drawn patented steel wire. That, in general, good micrographs can be obtained without a backing film is shown in Figs 8 and 9. Figure 8 gives the microstructure of high-carbon high-chromium tool steel in the hardened condition and Fig. 9 shows subboundaries and slip-lines in mild steel compressed 6 per cent after etching electrolytically.

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[6] Made by Smethurst High-Light, Ltd., Bolton, U. K.

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(3) D. E. Bradley, *British Journal of Applied Physics*, Vol. 5, pp. 65–66 (1954).

APPENDIX III

OBSERVATIONS OF BAINITE FORMATION WITH THE THERMIONIC EMISSION MICROSCOPE*

By Irwin I. Bessen[1]

In conjunction with studies by Subcommittee XI of ASTM Committee E-4, on AISI 4140 steel (see committee report in this volume) an examination of bainite formation in this alloy was undertaken. The technique used was that of thermionic emission microscopy; the aim was to provide data that could be later correlated with information derived from other independent techniques.

Description of the Method

Thermionic emission microscopy has been described by Burgers and Ploos van Amstel (**1**)[2] and by Heidenreich (**2**). Briefly, the specimen is made the source of electrons in an electron microscope. Emission current density is a function of the work function and the specimen temperature. The former, in turn, depends on the orientation of the crystalline lattice at the specimen surface and on the surface activation. Since the orientation varies from grain to grain, the grains can be distinguished by their brightness variations.

Two electromagnetic lenses are used to image the specimen on a fluorescent screen or 35-mm fine grain positive film. Pictures can be taken at intervals of one second or more.

The steel specimen was cut into a disk $\frac{3}{16}$ in. in diameter by about 0.02 in. thick. It was then spot welded to the end of a nickel cylindrical shell so as to form a cup with the specimen at the bottom. A 6-w tungsten heater positioned inside the cup served to heat the specimen by radiation.

Cooling was achieved primarily by radiation at an average rate of 90 F per sec. The temperature scale, determined previously on similar specimens of a high-purity eutectoid steel by observing the transformation temperatures, was accurate to within 40 deg.

Transformation at 950 F

Figure 1 shows the specimen is austenite at about 2370 F. The surface roughness which may be noted is the remaining evidence that plastic deformation has occurred on two previous austenite to bainite transformations. The originally polished surface shows the upheaval of the previously existent bainite needles. The white specks are oxidized barium activator particles.

Figures 2 to 6 were taken 10, 25, 28, 31, and 37 sec, respectively, after dropping the heater current. Figure 2, at a temperature greater than 950 F, shows that

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* Presented at a meeting of Subcommittee XI on Electron Metallography of ASTM Committee E-4 on Metallography, Atlantic City, N. J., June 19, 1956.

[1] Philips Electronics Inc., Mt. Vernon, N. Y.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 425.

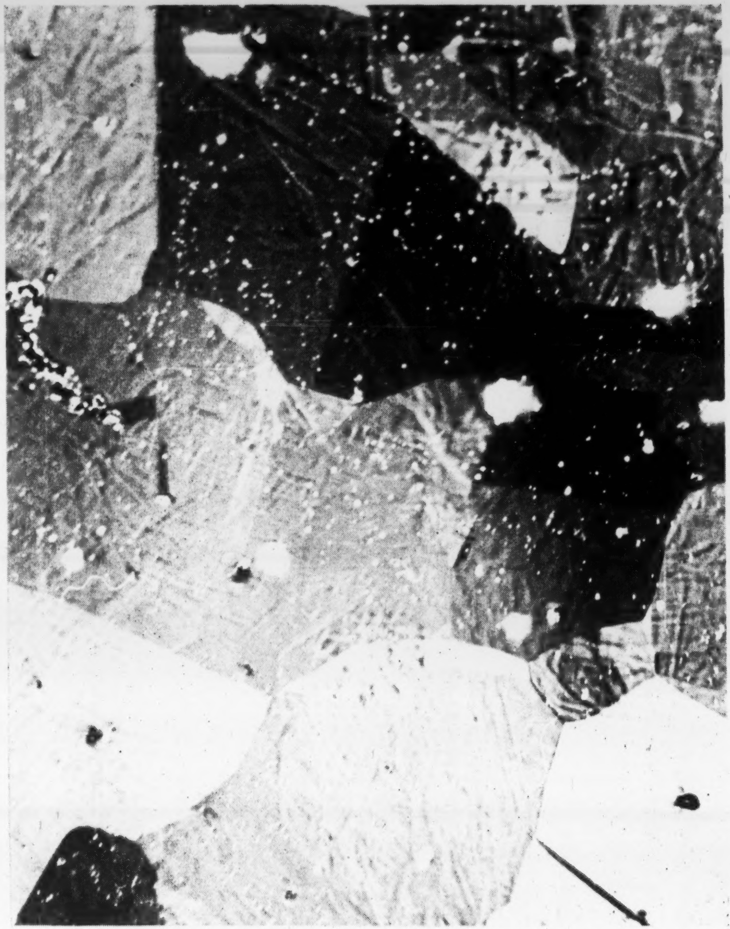

FIG. 1.—Austenite at 2370 F, Barium Activated (× 1000). Reduced one half in reproduction.

Rumpled surface shows strain relief caused by prior transformations. White specks are barium oxide.

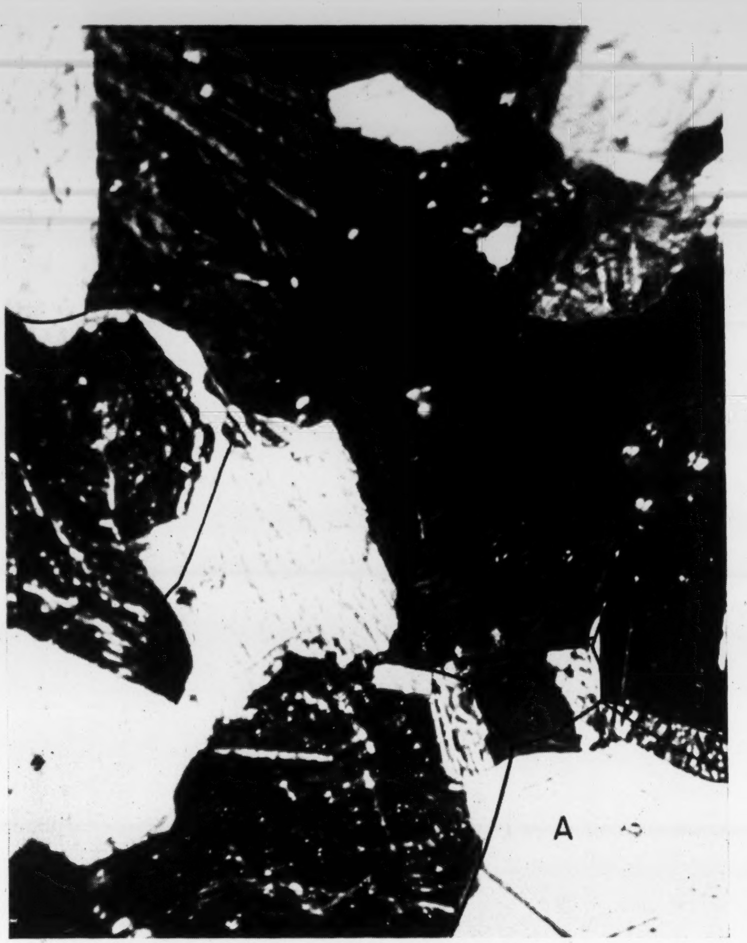

FIG. 2.—Supersaturated Austenite 10 sec After Dropping Temperature (× 1000). Reduced one half in reproduction.

Black lines show original austenite grain boundaries. Platelets appear in grain A. F appears to be ferrite. Rubidium activated.

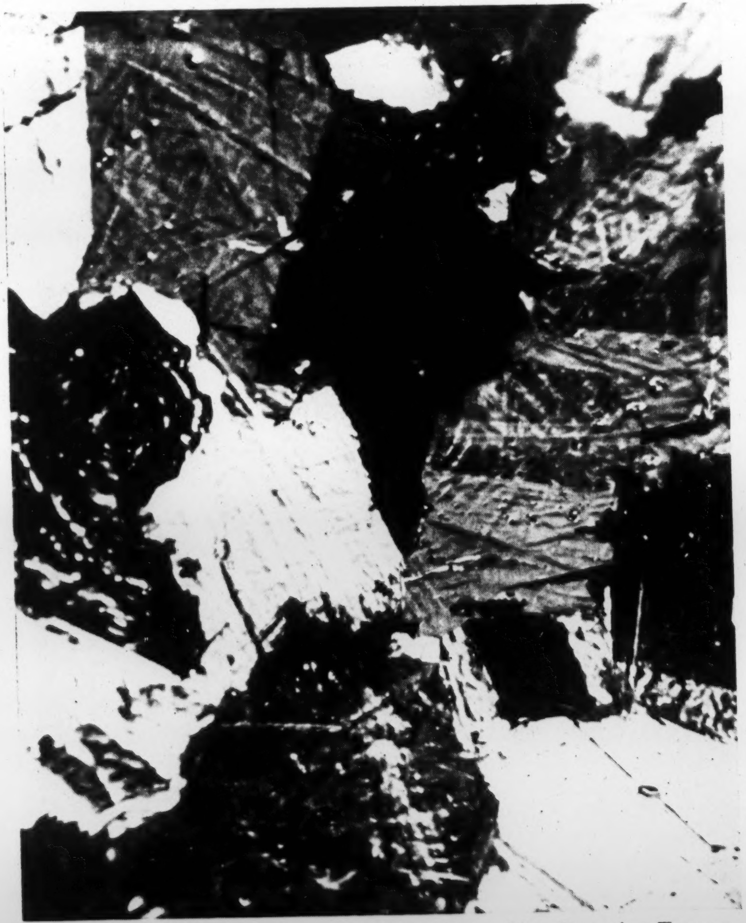

FIG. 3.—Bainite Nucleating in Austenite 25 sec After Dropping Temperature (× 1000). Reduced one half in reproduction.

Temperature is close to 950 F.

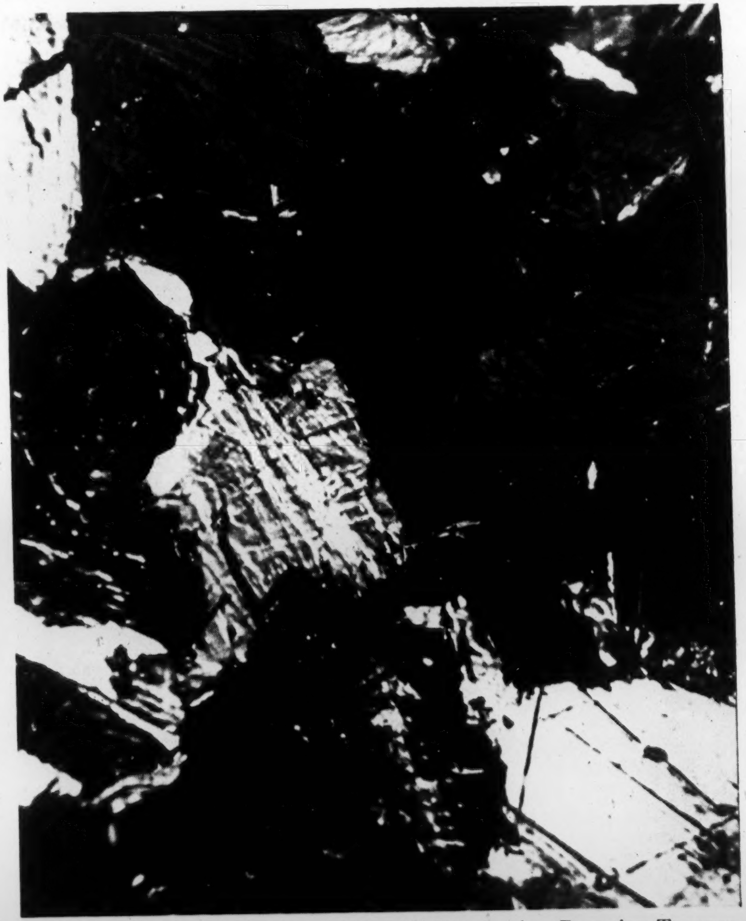

FIG. 4.—Bainite Nucleation and Growth 28 sec After Dropping Temperature (× 1000). Reduced one half in reproduction.

Intensity variations among photographs are caused by unequal activator application.

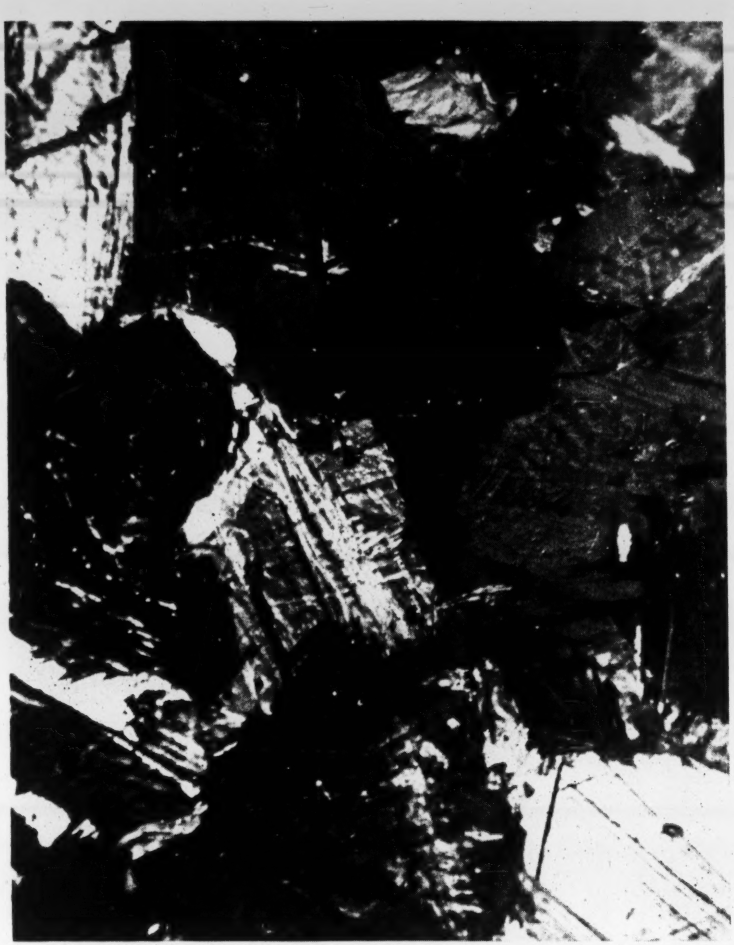

FIG. 5.—Bainite Nucleation and Growth 31 Sec After Dropping Temperature (× 1000). Reduced one half in reproduction.

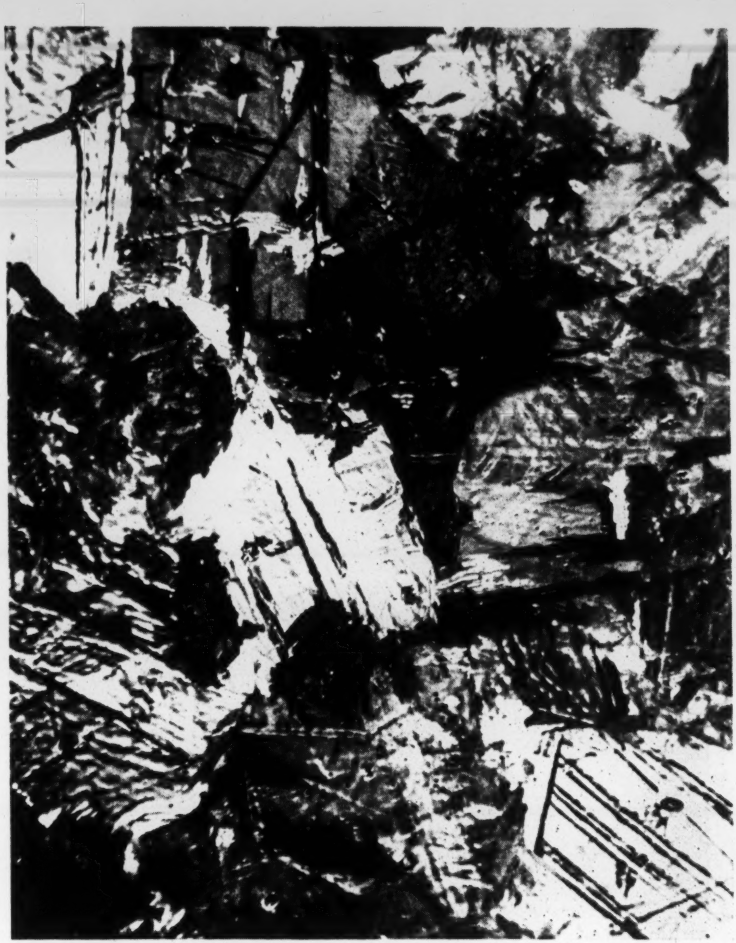

FIG. 6.—Bainite Nucleation and Growth 37 Sec After Dropping Temperature (× 1000). Reduced one half in reproduction.

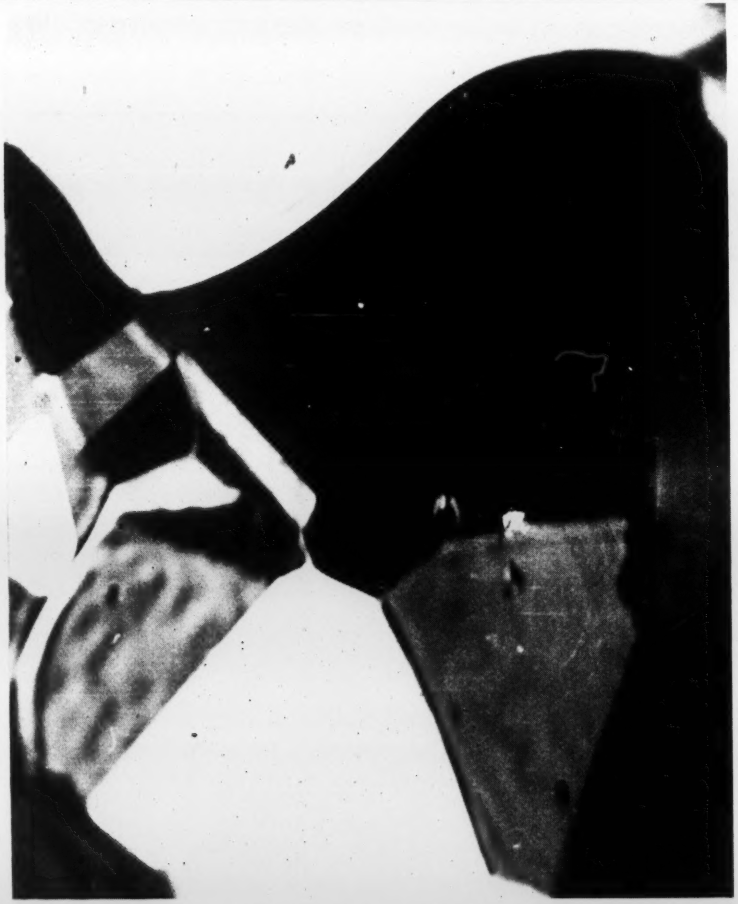

FIG. 7.—Austenite at 2200 F, Barium Activated (× 1000). Reduced one half in reproduction.

The field is shifted slightly relative to Figs. 8 to 12.

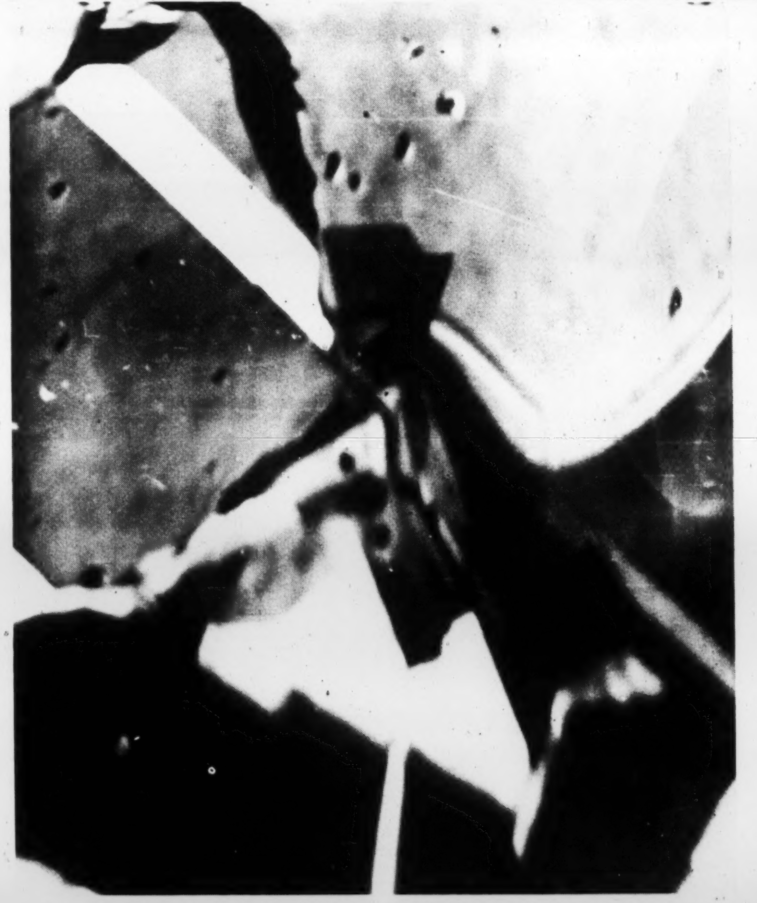

FIG. 8.—Ferrite and One or Two Bainite Needles Growing in Supersaturated Austenite at 1025 F About 20 Sec After Lowering Temperature. Cesium Activated (× 1000). Reduced one half in reproduction.

A prior austenite boundary parallels the existing one ahead of advancing bainite needle.

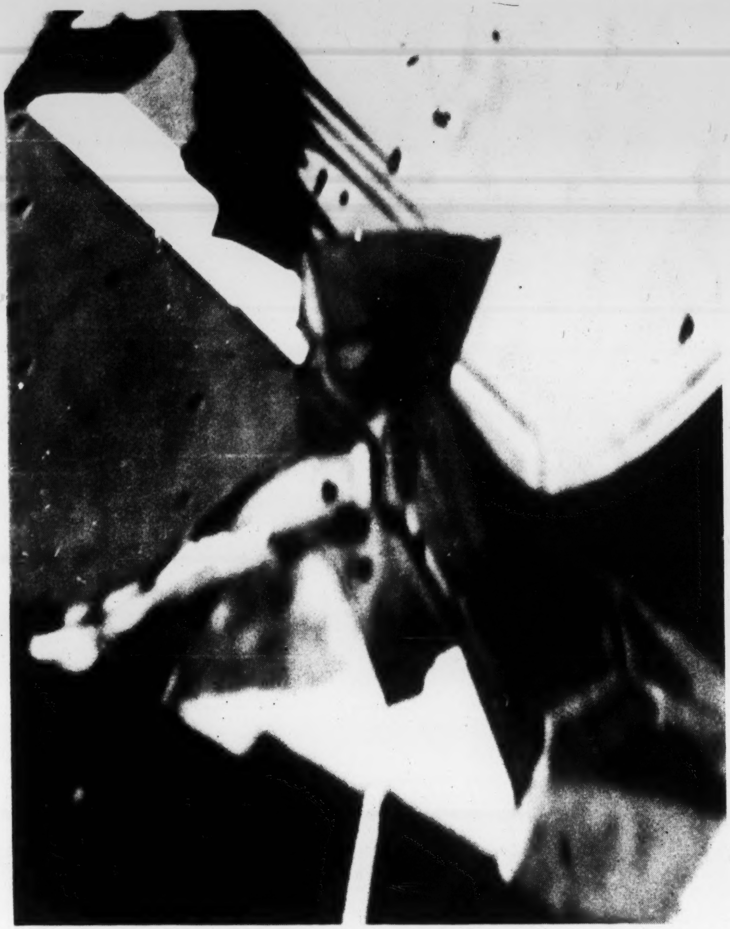

FIG. 9.—Growth of the Bainite Needle After Holding Isothermally at 1025 F for 23 Sec (× 1000). Reduced one half in reproduction.

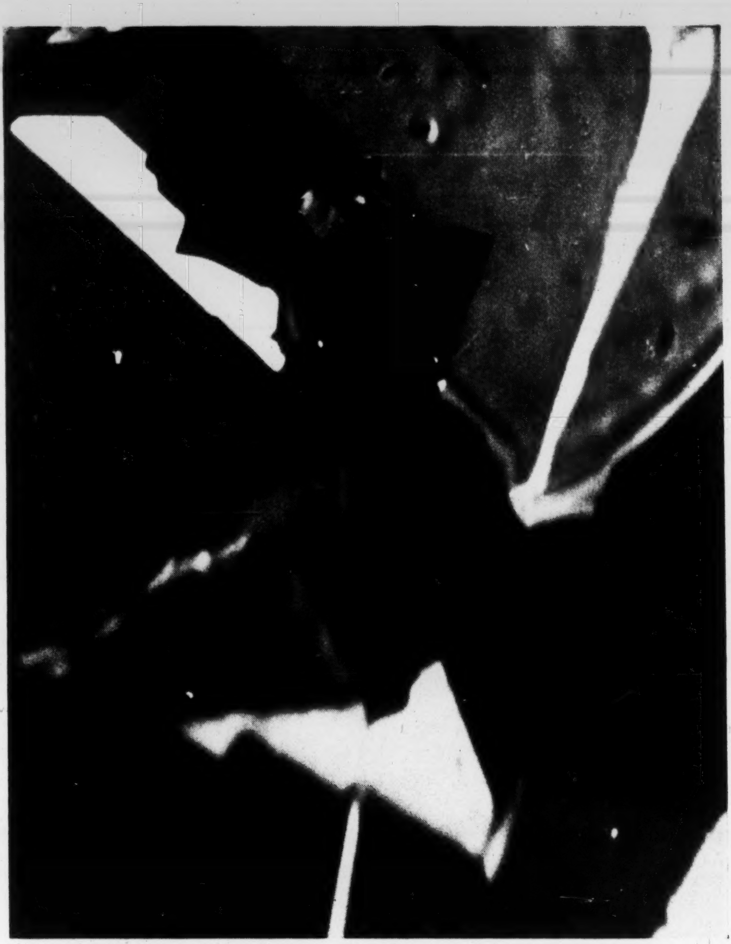

FIG. 10.—Bainite Needle Reaches Opposite Boundary and then Grows into Adjacent Grain in a New Direction, After Holding Isothermally at 1025 F for 26 Sec (× 1000). Reduced one half in reproduction.

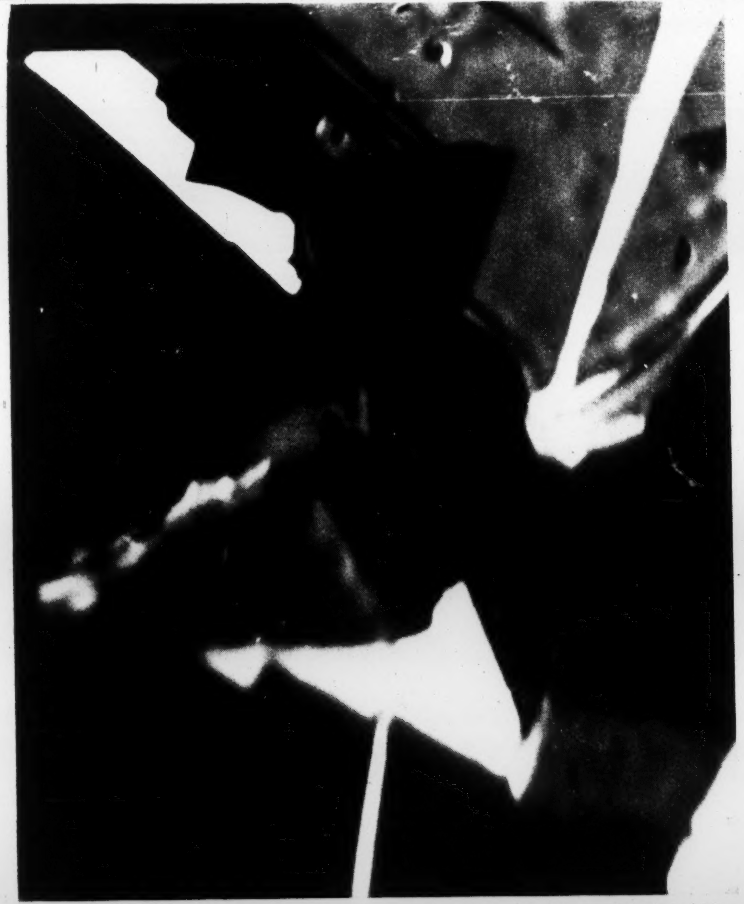

FIG. 11.—Bainite Grows into Adjacent Grain, and a Second Bainite Needle Approaches the First (× 1000). Reduced one half in reproduction.

Isothermally held at 1025 F for 35 sec.

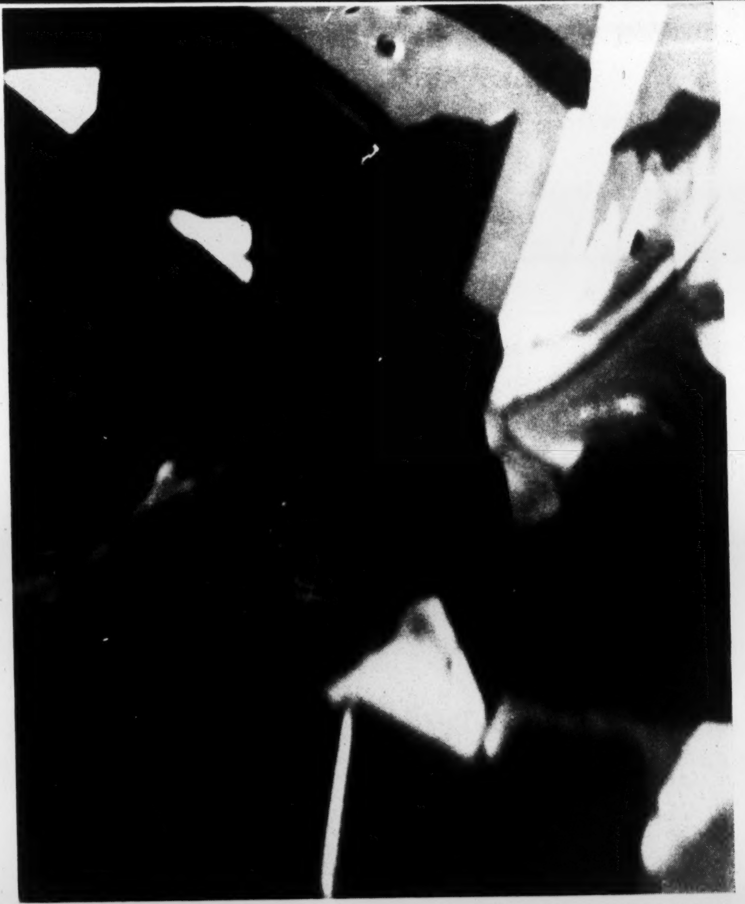

FIG. 12.—White Bainite Needle Grows in Opposite Direction in Original Austenite Grain. Black Needle is Terminated by the Edge of the White Needle Which has Subsequently Grown Laterally (× 1000). Reduced one half in reproduction.

Isothermally held at 1025 F for 53 sec.

grains *A* and *B* have been invaded by their surrounding grains, and grain *A* appears to be forming a plate-like precipitate. One set of platelets is directed the same as the austenitic twin. This austenite boundary migration was noted by Rathnau and Baas (3) who explained that the invaded grain has a high strain energy such that the more stable surrounding grains migrate their boundaries into the high energy region. If this is the case here, then the plate-like precipitate probably forms on a slip plane of the austenite.

Nucleation of bainite needles at boundaries and inhomogeneities appears at a later time than nucleation of the platelets (see Fig. 3). These needles seem to grow axially at a rate lower than the axial growth of the platelets, and higher than their own lateral growth (see Figs. 3 to 6). The platelets, once having generated to their full length, broaden thereafter. In all cases, the needles assume definite directions and do not cross grain boundaries. A maximum of four directions in any one austenite grain has been observed for the needle growth, and coupled with the fact that growth occurs parallel to the twinning planes, where twins are found, it appears that the family of planes of bainite propagation is the (111) set.

If the growth rate of the needles occurs by a diffusion process as one might expect (bainite discharging carbon into the austenite ahead of it and this diffusing away), the observed growth rates are not typical because surface diffusivity is greater than lattice diffusivity. Just how much of the detectable bainite grows along the surface and how much is grown primarily from under the surface is open to question. In any case, the propagation of the platelets at 950 F appears to be about five times greater than the axial growth of the needles.

TRANSFORMATION AT 800 F

We were not able to photograph bainite formed at 800 F, although its very rapid nucleation and growth have been observed. Almost all such bainite formed in platelets. Initially, the platelets appeared to propagate in a parallel array from one side of the grain towards the opposite side. When the platelets were more than half way across the grain, each platelet seemed to send out branches in a specific direction, branches from all original platelets being parallel. The result was the "feathered" structure typical of lower bainite.

TRANSFORMATION AT 1025 F

At 1025 F, very little bainite was observed. However, a sequence of two bainite needles growing slowly in the austenite was noted. These are shown in Figs. 7 to 12, in which most of the product phase is proeutectoid ferrite. One sees that the axial and lateral growths occur simultaneously here (though at different rates), and when the white needle reaches the grain boundary, it nucleates in the adjacent grain. The growth does not continue undeviated across the boundary as it does for pearlite (3), but it assumes a specific direction in the adjacent austenite grain.

APPEARANCE OF CARBIDES

Carbide formation in its earliest stages was not seen because of insufficient resolution. Carbides are first seen to appear two to three seconds after the bainitic needle forms. Figures 13, 14, and 15 show a sequence of bainite formation in which carbides can be seen. It is also possible to detect the prior austenite grain boundaries. It is seen that most of

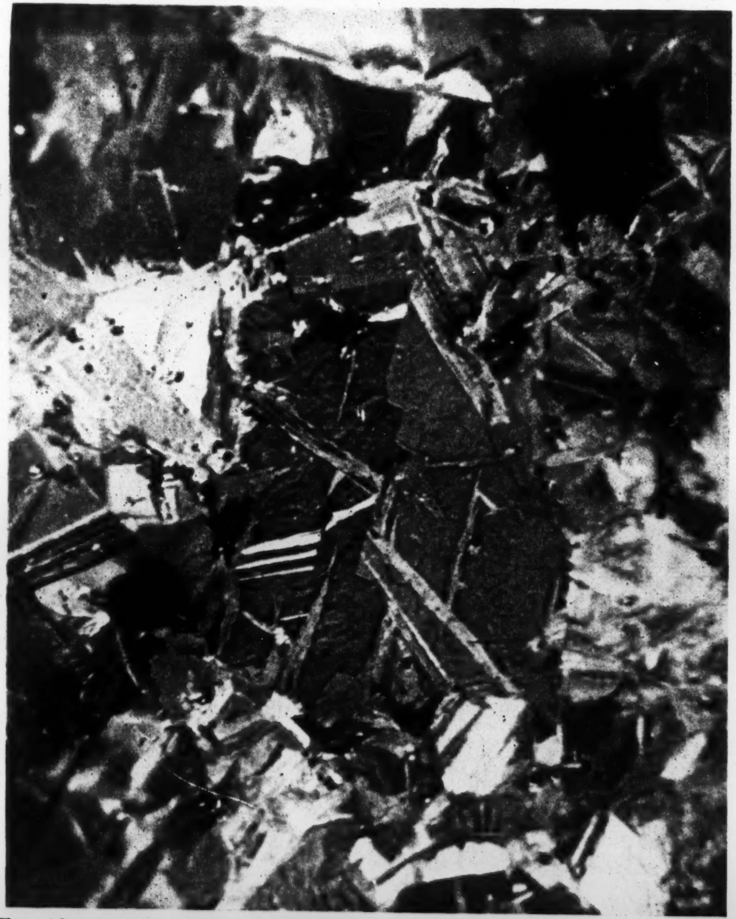

Fig. 13.—Bainite Forming in Supersaturated Austenite (× 800). Reduced one half in reproduction.

No carbides are visible after holding isothermally at 900 F for 25 sec.

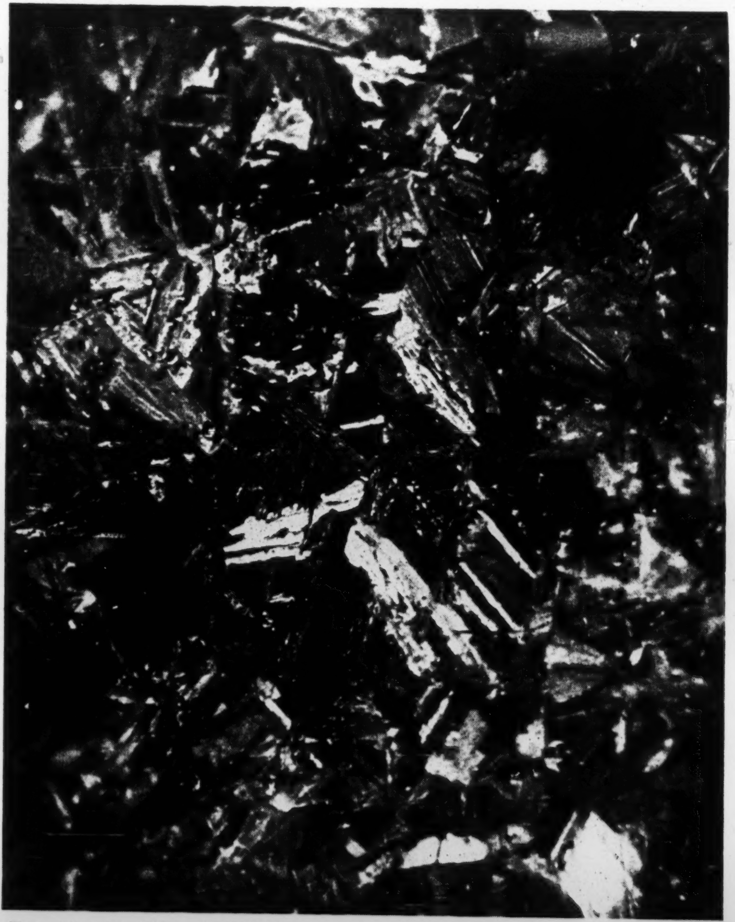

Fig. 14.—Carbides Are Well Developed in many Bainite Needles After Holding Isothermally at 900 F for 35 sec (× 800). Reduced one half in reproduction.

the carbides are oriented along the direction of a needle axis. Most of the other carbides are oriented in the direction of other needles in the same austenite grain.

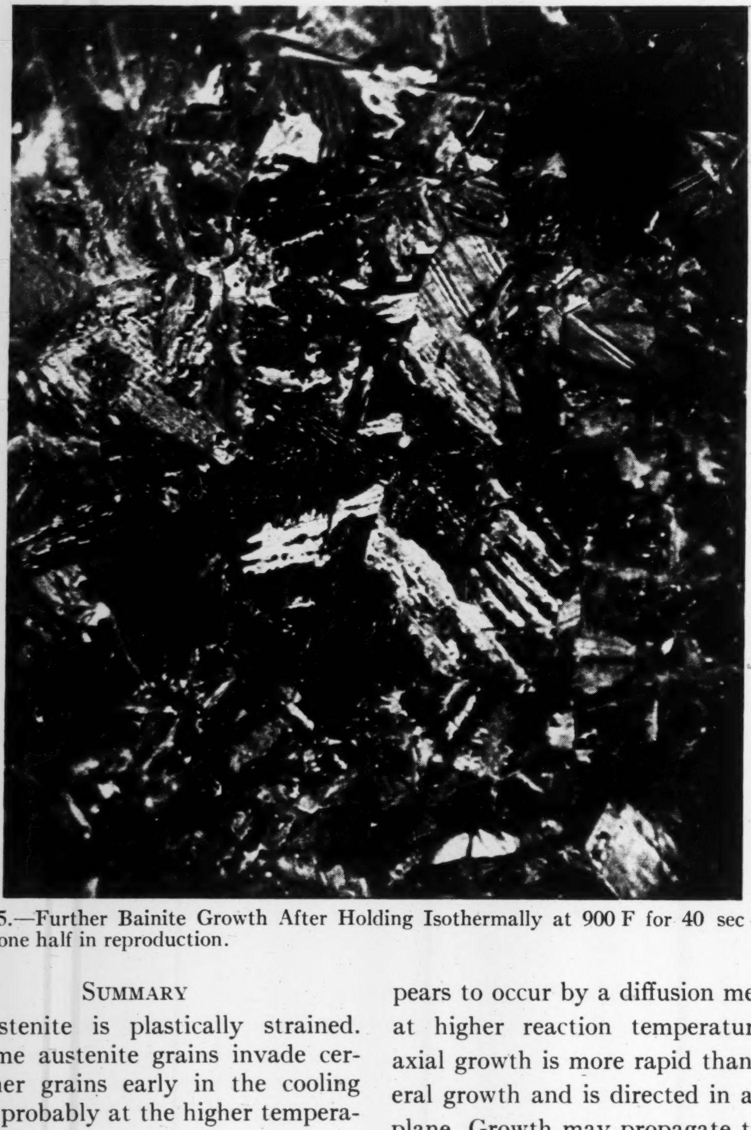

FIG. 15.—Further Bainite Growth After Holding Isothermally at 900 F for 40 sec (× 800). Reduced one half in reproduction.

SUMMARY

1. Austenite is plastically strained.

2. Some austenite grains invade certain other grains early in the cooling process, probably at the higher temperatures only.

3. Certain austenite grains show platelet precipitation and growth along definite planes. This mechanism is the predominant one at low reaction temperatures.

4. Growth of the bainite needles appears to occur by a diffusion mechanism at higher reaction temperatures. The axial growth is more rapid than the lateral growth and is directed in a definite plane. Growth may propagate to an adjacent grain of austenite after undergoing a change in direction.

DISCUSSION

The observations suggest the following:

1. Lattice strain in austenite is a prerequisite for bainite formation. Bainite may exist, as distinguished from proeutectoid ferrite and cementite, because the austenite grains of lower energy cannot accommodate the highly strained regions by boundary migration at lower temperatures.

2. The driving force for bainite formation has two components: the lattice strain and the chemical or solubility driving force. Both depend on the reaction temperature. In low-temperature reactions, the strain is high, diffusion is low and a platelet structure appears, typical of lower bainite. In higher temperature reactions strain is lower but still exists; diffusion is rapid.

3. Whether super-saturated ferrite forms first, subsequently throwing out carbide to form bainite, or whether ferrite and carbide precipitate together cannot be determined from these observations.

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(2) R. D. Heidenreich, "Thermionic Emission Microscopy of Metals, Part I. General," *Journal of Applied Physics*, Vol. 26, No. 6, June, 1955, p. 757.

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APPENDIX IV

ELECTROPOLISHING OF FERROUS METAL SPECIMENS FOR ELECTRON METALLOGRAPHY*†

By F. W. Boswell[1]

In the electrolytic method of polishing, the metal specimen is made the anode of an electrolytic cell, and the surface material is removed by electrochemical action under conditions such that the surface becomes smooth. Ideally the method should be well suited for electron metallography since a surface can be prepared which is entirely free from the mechanical disturbances associated with cutting, grinding, and mechanical polishing. However electropolishing may be subject to experimental limitations with the result that mechanical polishing has been used much more extensively. The possible limitations are as follows:

1. Electrolytes may be dangerous to handle.
2. The surface may be etched as a result of electropolishing.
3. Results may not be reproducible (this is related to 2 above).
4. Elaborate equipment may be required.
5. Time required to obtain a suitable polish may be too long.
6. The surface may be wavy rather than flat.
7. A particular electrolyte may not be applicable to all ferrous alloys.
8. Inclusions may be pitted out and cracks exaggerated.

In order to find a method of electropolishing which minimized the above limitations an investigation of the various electrolytes that have been recommended for ferrous specimens was undertaken, excepting those solutions involving perchloric acid which were considered somewhat hazardous for general laboratory use. Of the various electrolytes investigated, all except one produced surfaces in an etched condition. Such etches are generally variable and highly undesirable for electron metallography where precise control of etch depth is essential for optimum results. The chromic acid - acetic acid electrolyte recommended by Morris (**1**)[2] is the exception mentioned above. This method produced surfaces which were practically etch free and hence the etch depth could be carefully controlled in a subsequent operation. Limitations 1 to 6 above are negligible when this electrolyte is used, since, in addition to producing a high polish, relatively simple equipment may be used, polishing times are reasonable, surface waviness is slight, and the method is applicable to a wide variety of ferrous alloys and microstructures. Since limitations 7 and 8 are only of significance in special cases, the method appears to be very suitable for most ferrous electron metallography. In this paper the method is outlined, and results of an electron microscopic examination of electropolished surfaces of several typical ferrous microstructures are presented.

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* Presented at a meeting of Subcommittee XI on Electron Microstructure of ASTM Committee E-4 on Metallography, Atlantic City, N. J., June 19, 1956.

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[1] Physical Metallurgy Div., Mines Branch, Dept. of Mines and Technical Surveys, Ottawa, Ont., Canada.

[2] The boldface numbers in parenthesis refer to the list of references appended to this paper, see p. 435.

EXPERIMENTAL METHOD

Our method is an adaptation of that recommended by Morris (1). The composition of the electrolyte is as follows:

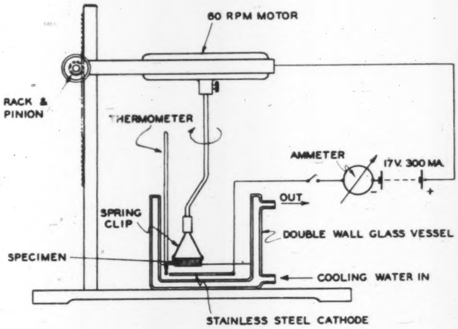


FIG. 1.—Diagrammatic Sketch of Electropolishing Apparatus.

Glacial acetic acid (99.5 per cent by weight minimum)	133 ml
CrO_3	25 g
Water	7 ml

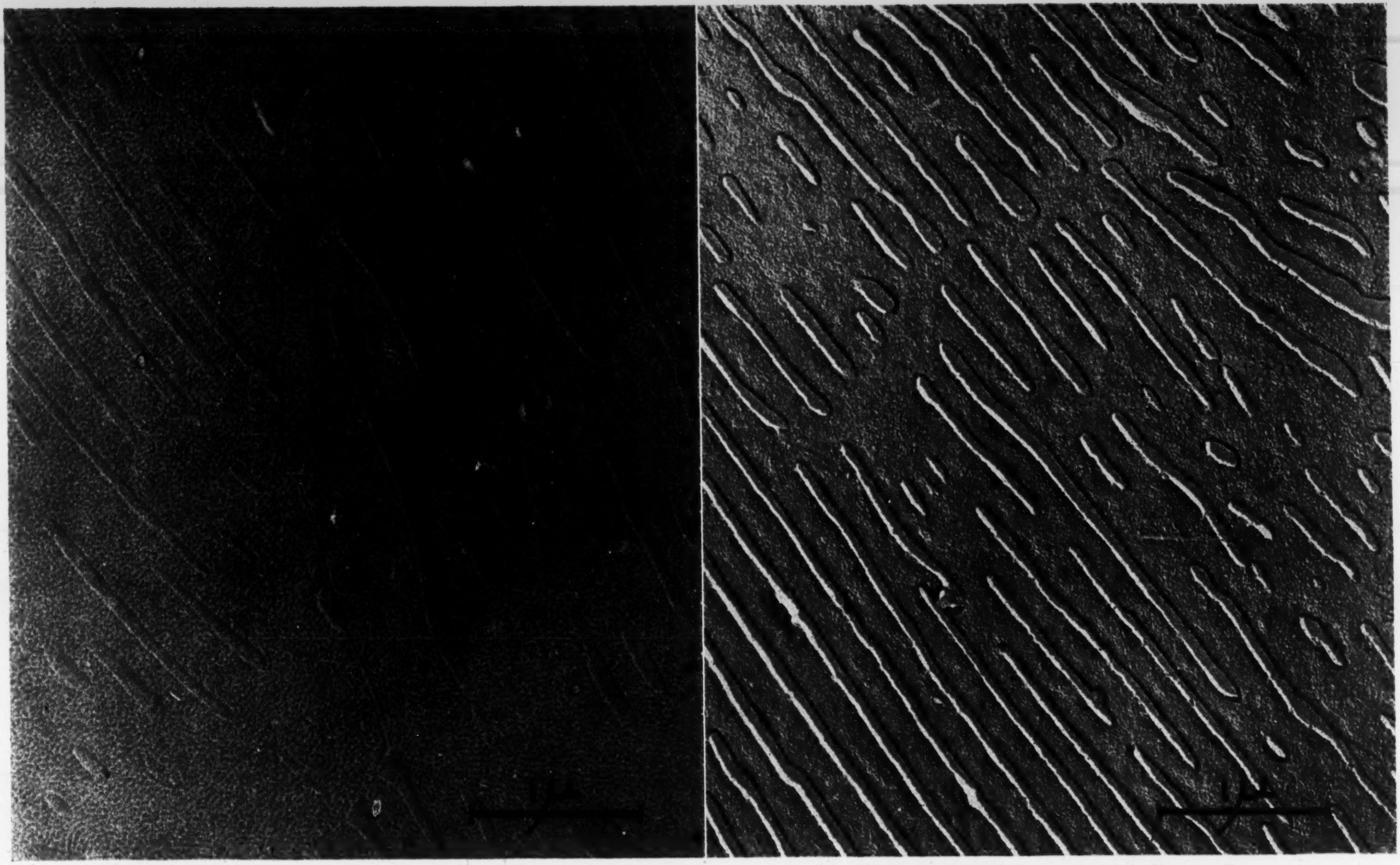
The chromium trioxide will take several hours to dissolve at room temperature and solution may be hastened by heating the mixture in a bath at 60 to 70 C. The paper of Morris (1) should be consulted for details and precautions on mixing the electrolyte. The apparatus used is shown in Fig. 1. The specimen is first ground on the usual metallographic papers down to a 000, and is then mounted in the spring clip so that the ground surface is parallel to the cathode. The electrolyte should be kept within the temperature range of 17 to 20 C during use. Spacing between the specimen and the cathode should be approximately $\frac{1}{4}$ in. It has been found that an operating voltage of 17.5 v and a current density of about 0.40 amp per sq cm gives good results with polishing times of 5 to 10 min. As soon as it is removed from the electrolyte, the specimen is rinsed in a 50:50 acetone:methyl alcohol solution and dried from benzene. After drying, the surface of the metal is very reactive and should be etched immediately.

The electrolyte deteriorates during use and generally lasts about one week. An exhausted electrolyte will result in a poorly polished surface or in difficulty in etching apparently arising from a passivating surface film. This condition in the electrolyte appears quite suddenly and as soon as it is detected the solution should be discarded.

EXPERIMENTAL RESULTS

In Figs. 2 to 6 comparison replicas are shown of five typical ferrous microstructures in the "as polished" and etched conditions. Etching was carried out in a fresh solution of 1 per cent picric acid in methanol. Replicas were made of 0.5 per cent parlodion in amyl acetate, unless otherwise noted, and backed with a thick film of "Elvanol." All replicas were shadowed with chromium.

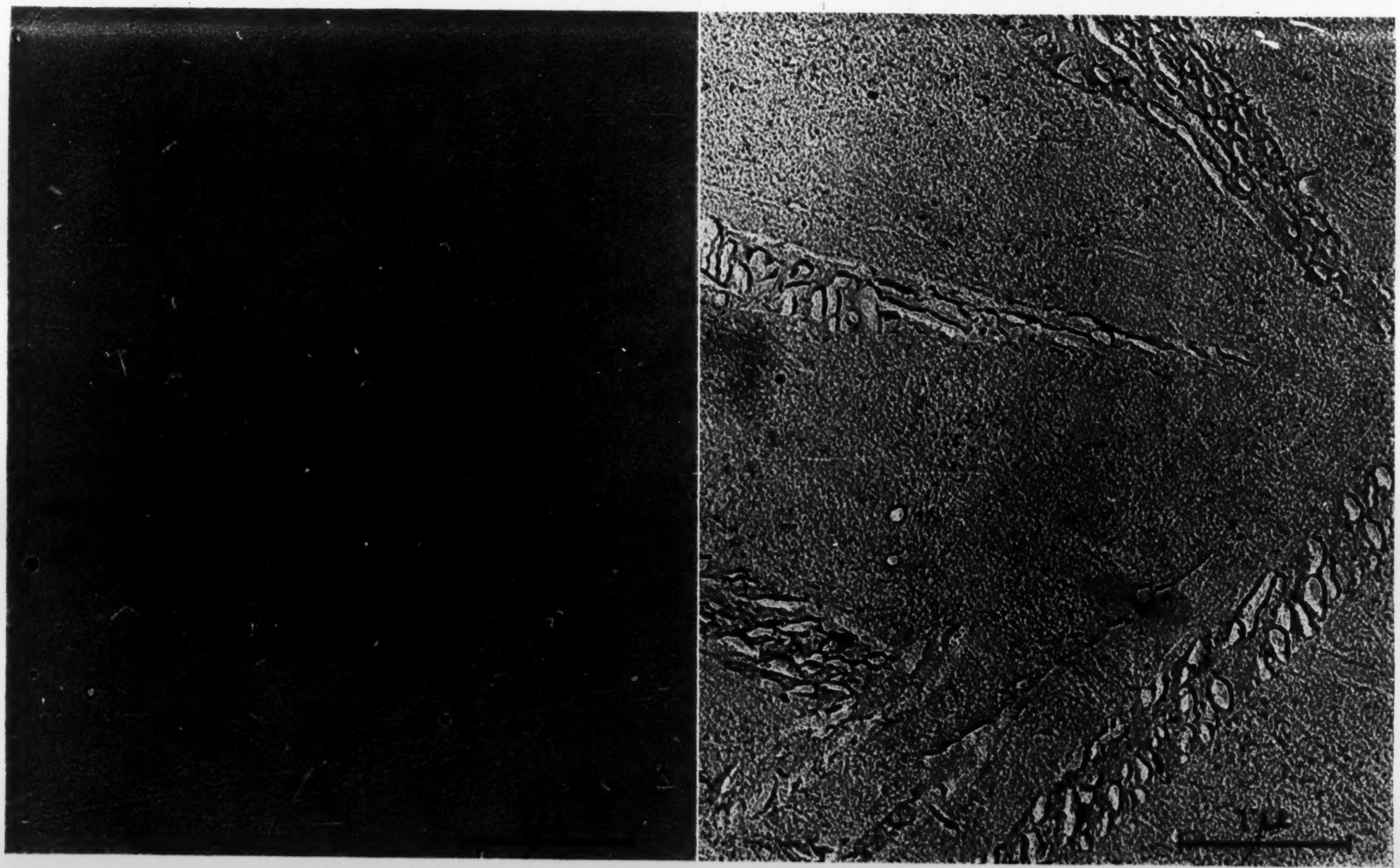
Figure 2 shows electron micrographs of coarse pearlite in a plain carbon eutectoid steel. In Fig. 2(*a*), the carbide lamellae are in relief by somewhat less than 100 Å and appear smooth to better than 100 Å. In Fig. 2(*b*) the etch depth is about 300 Å. The slight relief remaining after electropolishing is thus negligible compared to the etch depth normally used for electron metallography. In Fig. 3 are seen electron micrographs of eutectoid steel specimens partially transformed to lower bainite at 570 F



(*a*) As polished, 0.5 per cent parlodion replica. Note "orange peel" structure on ferrite.

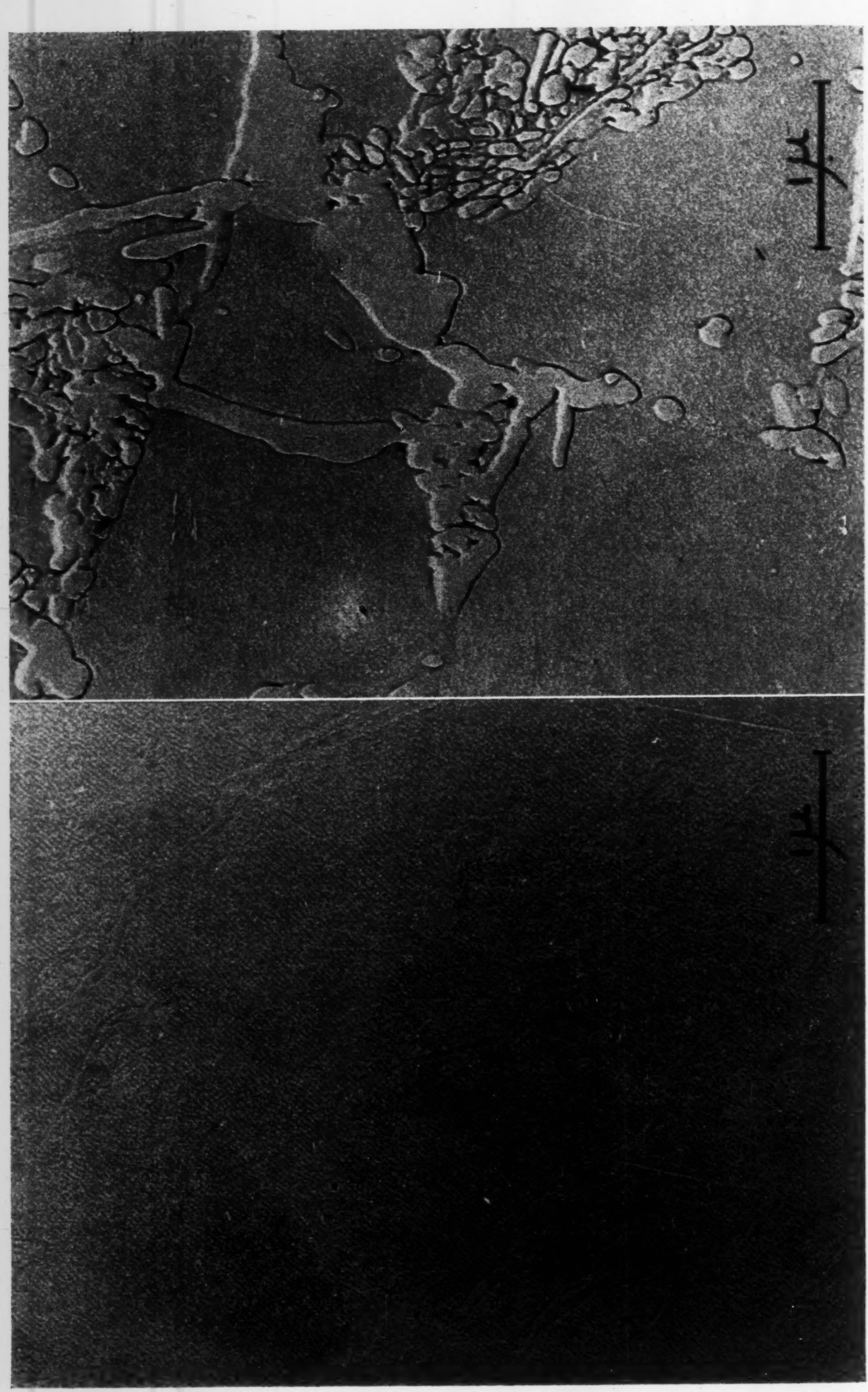
(*b*) Etched, 0.75 per cent parlodion replica.

FIG. 2.—Electron Micrographs of Coarse Pearlite in Eutectoid Steel.
Shadowed with chromium at 45 deg.



(*a*) As polished. (*b*) Etched.

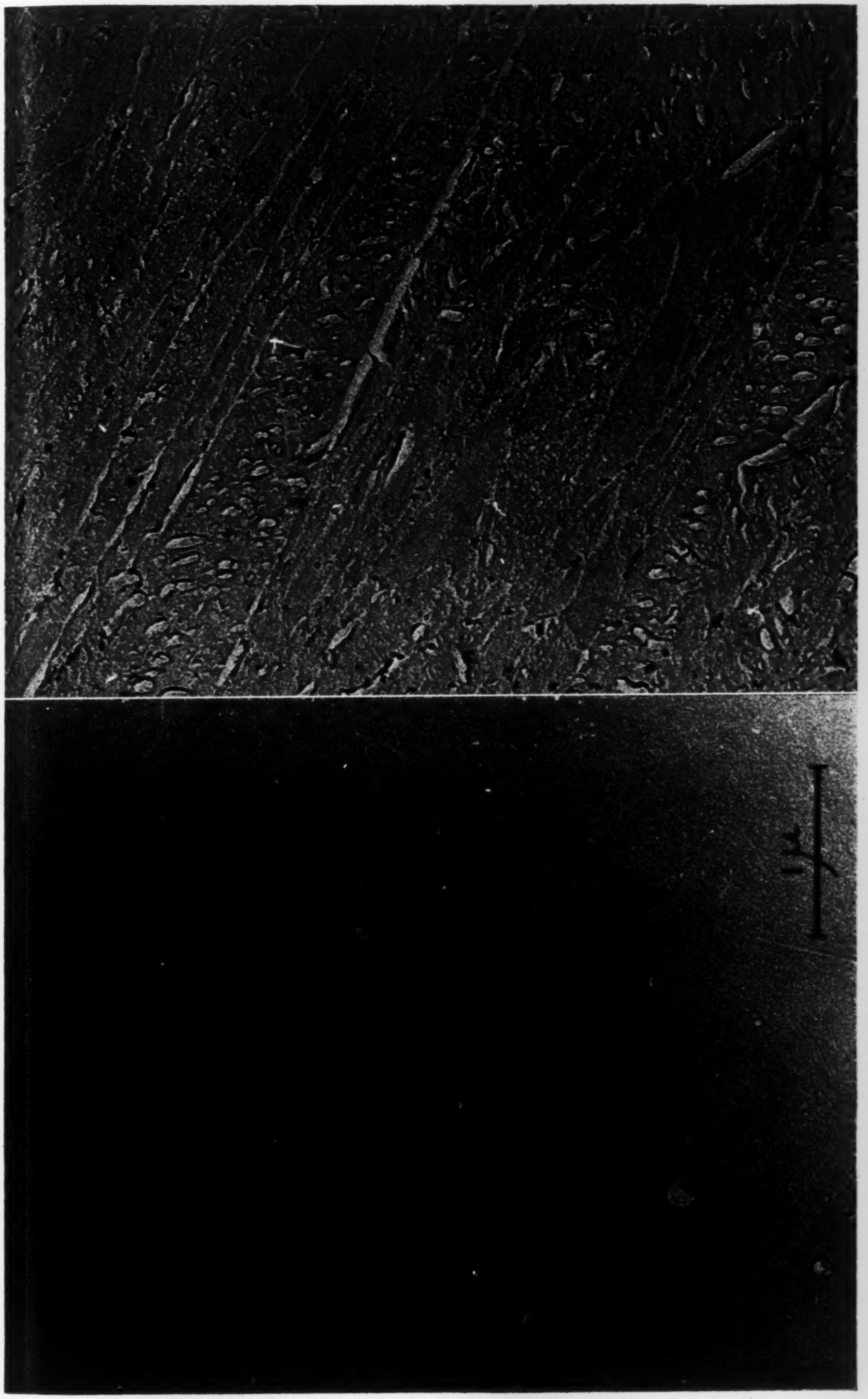
FIG. 3.—Electron Micrographs of Lower Bainite and Martensite in Eutectoid Steel. Shadowed with chromium at 45 deg.



(a) As polished. (b) Etched.

FIG. 4.—Electron Micrographs of AISI 4140 Steel Transformed 5 hr at 900 F.

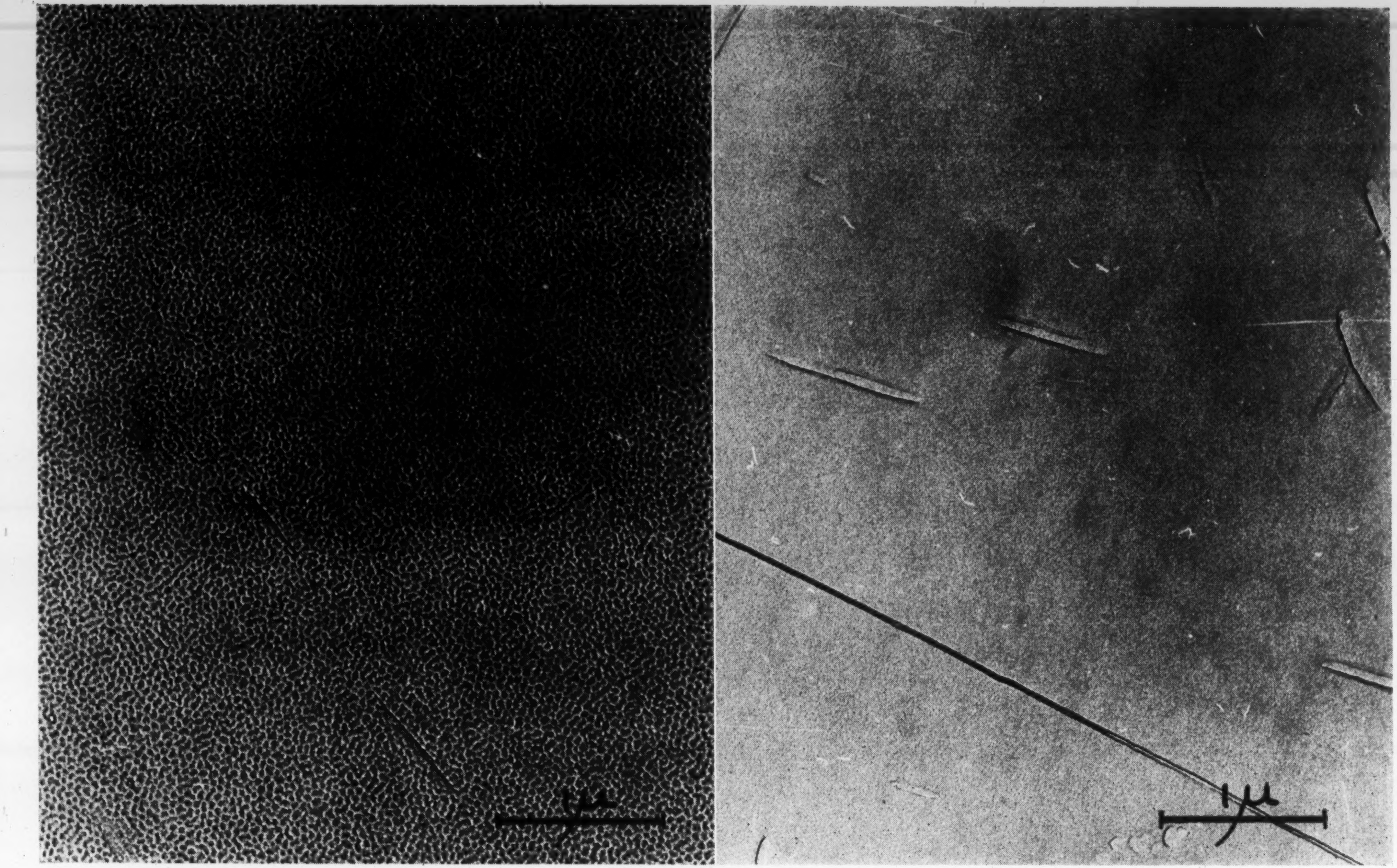
Shadowed with chromium at 45 deg.



(*a*) As polished. (*b*) Etched.

FIG. 5.—Electron Micrographs of AISI 4140 Steel Transformed 1 hr at 400 F.

Shadowed with chromium at 27 deg.



(*a*) As polished. Note "orange peel" structure. (*b*) Etched.

FIG. 6.—Electron Micrographs of High Purity Alpha Iron Containing 0.01 per cent Carbon, Aged at 392 F for 2 hr. Shadowed with chromium at 45 deg.

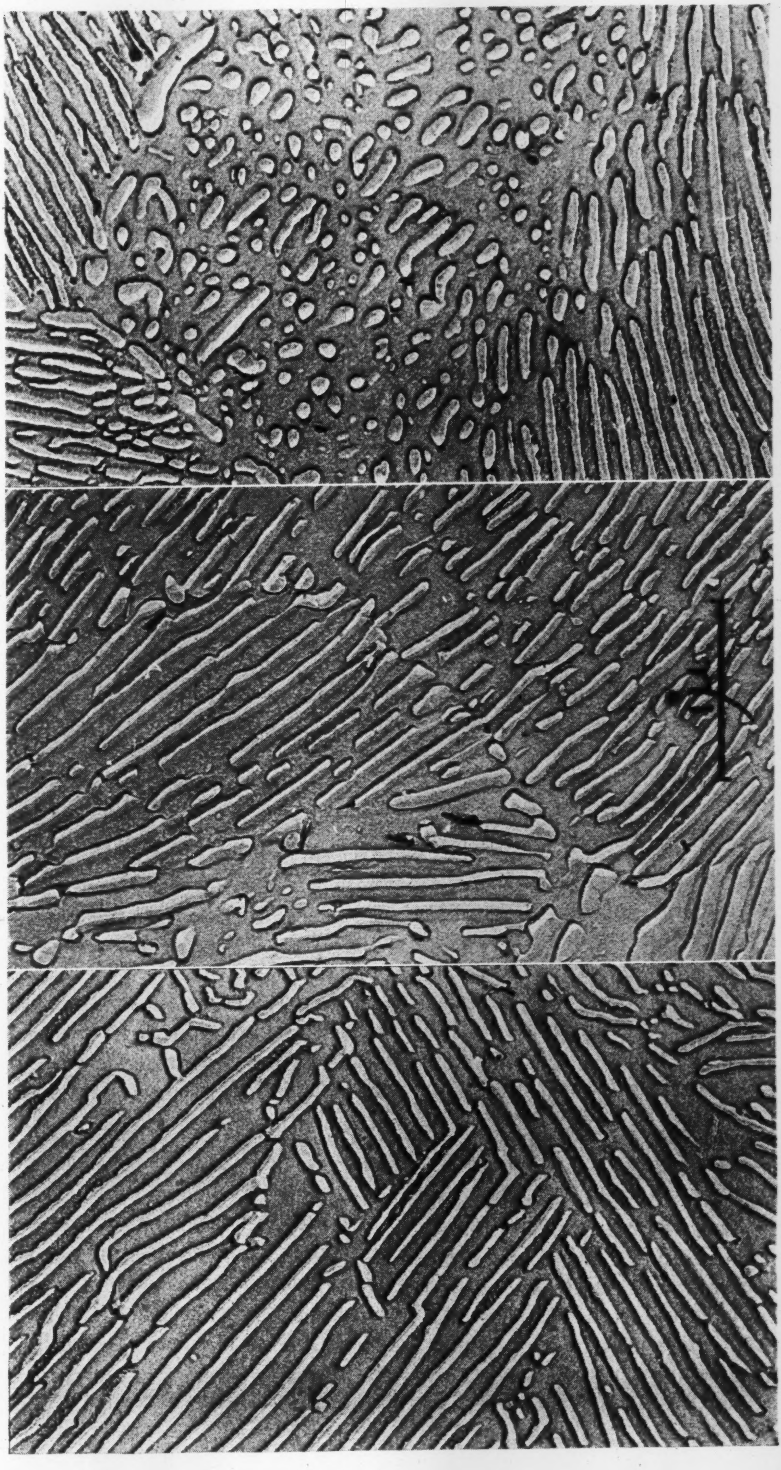


FIG. 7.—Electron Micrographs of Three Specimens of Fine Pearlite in Eutectoid Steel.

Each Specimen was Electropolished and Etched for 20 sec. Replicas of 0.75 per cent parlodion in amyl acetate and shadowed with chromium at 45 deg.

and then quenched. In Fig. 3(*a*) the martensitic region is smooth to about 100 Å and there is a barely detectable relief effect associated with the carbide particles in the bainite needles. As in the above case the etch depth in Fig. 3(*a*) is negligible compared to that in Fig. 3(*b*).

Electron micrographs of AISI 4140 steel transformed 5 hr at 900 F (ASTM Subcommittee XI of E-4 specimen No. 56) are presented in Fig. 4. In Fig. 4(*a*) the relief is so slight that the structure is barely visible. Figure 5 shows electron micrographs of the same steel (ASTM Subcommittee XI of E-4 specimen No. 59) after transforming for 1 hr at 400 F. After polishing (Fig. 5(*a*)) only a slight background roughening at the 100 to 200 Å level is visible.

In Fig. 6 are shown electron micrographs of high-purity alpha iron containing 0.01 per cent carbon. The specimen was quenched and aged at 392 F for 2 hr. In Fig. 6(*a*) it will be seen that the surface has the "orange peel" structure that may also be noted on some of the preceding micrographs, and the carbide platelets may be detected in relief. The "orange peel" effect may be a roughness of the metal itself or it may be the structure of a film resulting from electropolishing. In any case it is not detected after etching (Fig. 6(*b*)). Although the individual undulations of the "orange peel" structure are 300 to 400 Å in lateral dimensions, the variation in elevation appears to be less than 100 Å. We therefore feel that structural detail on the 100 Å level should be detectable in electropolished and lightly etched ferrite.

Figure 7 is an attempt to indicate the reproducibility that may be attained with this method of surface preparation. Three specimens of fine pearlite in eutectoid steel were electropolished and etched for 20 sec in a fresh solution of 1 per cent picral in methanol. Replicas were prepared from 0.75 per cent parlodion in amyl acetate. It may be seen that the etch depth is nearly identical on each specimen.

TABLE I.—COMPARISON OF ADVANTAGES AND DISADVANTAGES OF MECHANICAL AND ELECTROLYTIC POLISHING USING A CHROMIC-ACETIC ACID ELECTROLYTE.

Factor Considered	Mechanical Polishing	Electrolytic Polishing
Hazard	Negligible	Negligible
Surface micro-smoothness	Smooth	Very slight etch may occur
Reproducibility	Good	Good
Surface flatness	Very flat	Some waviness
Cold work at surface	Disturbed layer of variable thickness	Surface not disturbed
Etching characteristics	May depend upon worked layer	Uniform
Operator	Must be skilled and experienced	Extensive experience not required
Equipment	Standard	Easily assembled
Polishing time	15 min	10 to 15 min
Inclusions	Retained	May be pitted
Range of application	Very general	Some limitations

DISCUSSION

The results published in the reports of Subcommittee XI of Committee E-4 have demonstrated that careful mechanical polishing by skilled metallographers is suitable for electron metallography of a wide variety of microstructures found in eutectoid steel. However, mechanical polishing has the inherent disadvantage that it leaves a cold-worked layer at the surface which may deform the microstructure. In practice, this layer is made thinner and thinner by alternate polishing and etching until the final very thin layer of disturbed metal is removed in the final etch. The depth of the final disturbed film of metal depends to some extent both upon the specimen hardness and the skill of the metallographer, but it appears that for microstructures occurring in eutectoid steel an etch depth of 200 to 300 Å is sufficient to remove the disturbed metal in a properly prepared specimen (2). In general the etch depth must be less than the largest dimension of the precipitate particles under examination. Hence as replica techniques are improved to provide better contrast and resolution it appears that lighter etching will be employed for the examination of very fine precipitate particles, and mechanical disturbance of the surface layer will become increasingly undesirable. The problem is accentuated for very soft specimens. It is therefore likely that electrolytic methods of specimen preparation will be used more extensively in the future. A summarized comparison of the two methods of polishing is given in Table I.

CONCLUSIONS

1. The chromic acid - acetic acid electrolyte proposed by Morris (1) is suitable for the preparation of plain carbon steels, low alloy steels (AISI 4140) and alpha iron for electron metallography.

2. In the coarser microstructures there may be a slight etch present after electropolishing, but the depth of the etch is always much less than that suitable for electron metallography.

3. Some structures, after electropolishing, possess an "orange peel" structure. It is unlikely that this effect would interfere with detection of structural details in the 100 Å range after suitable etching.

4. Reproducible results are obtained when the specimen is etched immediately after electropolishing, provided that the electrolyte is not too old.

REFERENCES

(1) C. E. Morris, "Electropolishing of Steel in Chrome-acetic Acid Electrolyte," *Metal Progress*, Vol. 56, p. 696 (1949).

(2) W. L. Grube and S. R. Rouze, "Specimen Polishing Techniques for Electron Metallography of Steel," Symposium on Techniques for Electron Metallography, Am. Soc. Testing Mats. (1954). (Issued as separate publication *ASTM STP No. 155*.)

APPENDIX V

COMPARISON OF SURFACE AND VOLUME TRANSFORMATIONS IN ALLOY STEEL*

By L. S. Birks[1] and R. T. Seal[1]

In studying phase transformations by high-temperature X-ray diffraction or by the thermal emission electron microscope, one should know how well the transformation near a free surface approximates the transformation taking place in the volume of the material. For instance, the difference in physical constraint near the surface may affect some reactions such as bainite or martensite. This paper represents a preliminary comparison of the surface and volume bainite transformations in two alloy steels.

Experimental Results

Table I shows the composition of the two alloy steels. Both of them have bainite transformation regions that extend over several hundred degrees Fahrenheit. Replicas for the electron microscope were prepared from specimens that had been heat treated in vacuum in the high-temperature X-ray diffractometer previously described by one of the authors.[2] Prior to the heat treatment, the specimens were polished metallographically so that the free surface could be observed after transformation.

Nickel-Chrome Steel:

First consider the appearance of the unetched surface; in general it is roughened due to plastic yielding during transformation. Figure 1 shows the surface of the nickel-chromium steel that had been isothermally transformed at 850 F for 5 min to give bainite. The prior austenite grain boundaries are apparent along with fine parallel lines that have a terraced appearance but are obviously not sliplines because of their curved nature. It should be noted that there is no recognizeable bainite structure in the sense that no carbides are visible. This means that in the emission electron microscope, the appearance of carbides is due to difference in work function rather than physically outlined carbide particles. That the bainite structure exists at the surface is illustrated in Fig. 2 (*a*) wherein

TABLE I.—ALLOY COMPOSITION, PER CENT.

Designation	Carbon	Nickel	Chromium	Manganese	Molybdenum	Silicon
Nickel-Chromium Steel	0.3	2.8	1.25	0.22	0.10	0.10
AISI 4140	0.4	...	1.0	0.8	0.14	0.3

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* Presented at a meeting of Subcommittee XI on Electron Microstructure of ASTM Committee E-4 on Metallography, Atlantic City, N. J., June 18, 1956.

[1] Electron Optics Branch, Optics Div., U. S. Naval Research Lab., Washington, D. C.

[2] L. S. Birks, *Review of Scientific Instruments*, Vol. 25, p. 963 (1954).

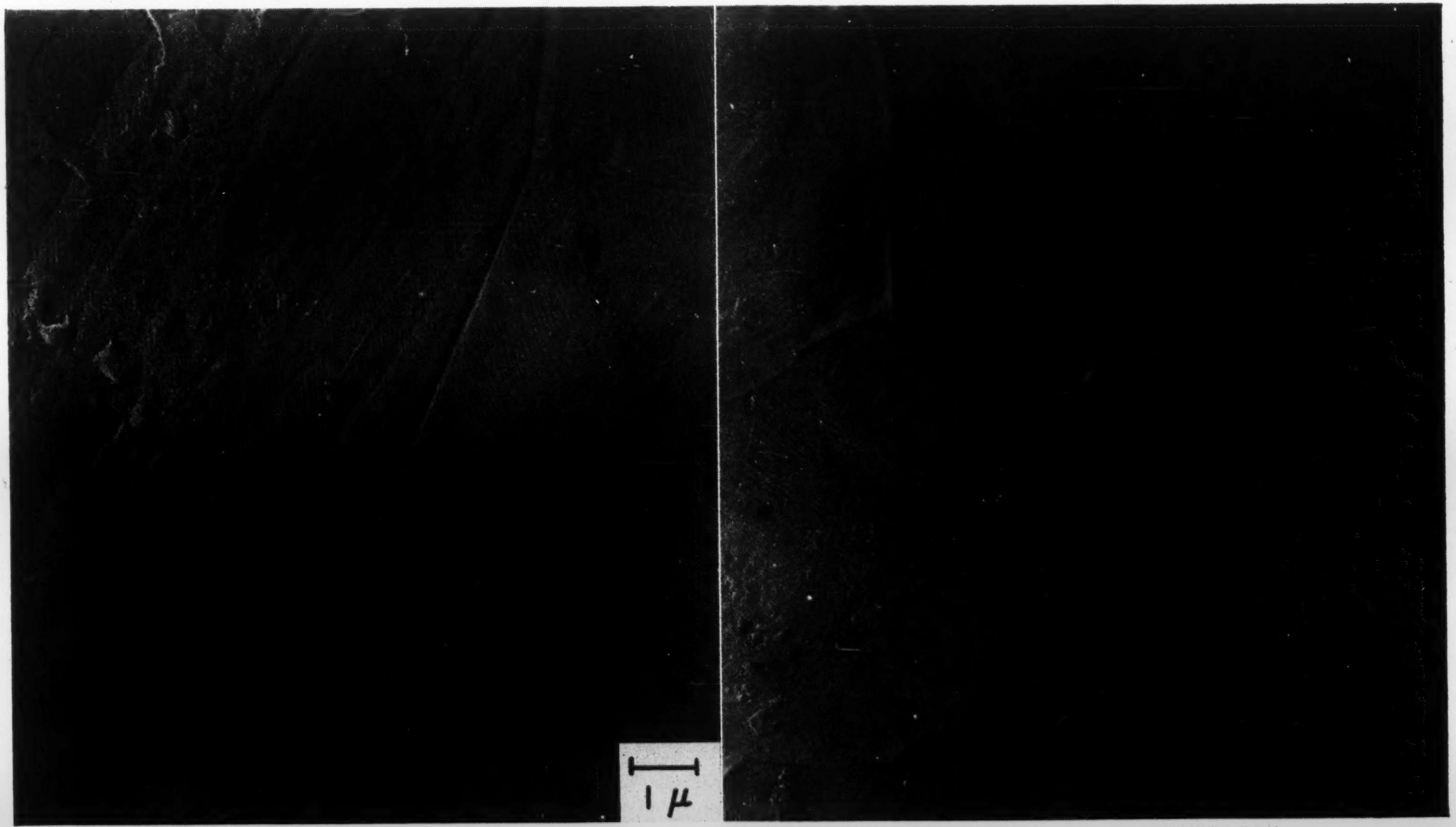

FIG. 1.—Unetched Surface of Nickel-Chromium Steel; Austenitized at 1600 F, Isothermally Transformed at 850 F for 5 Min.

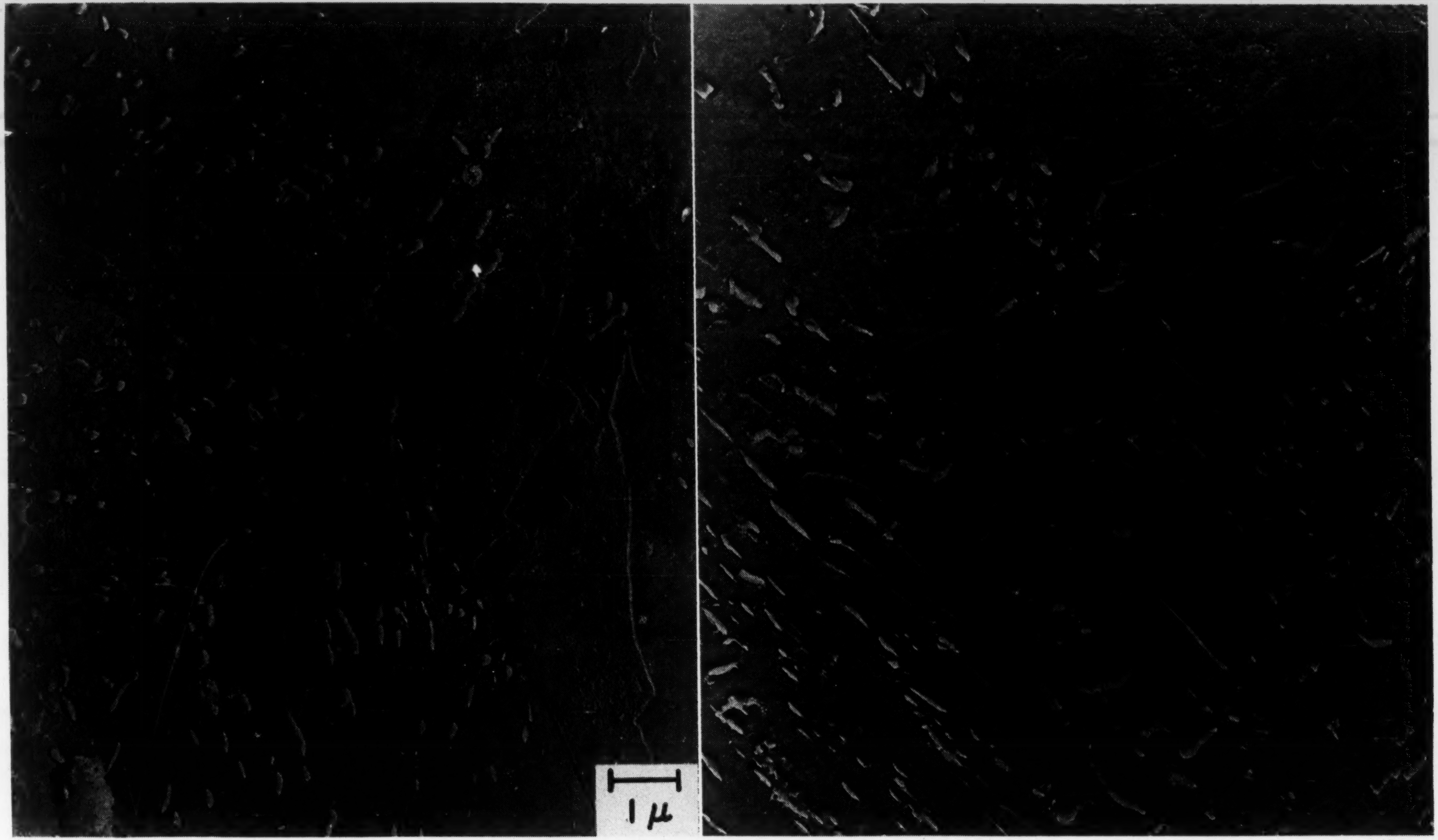

FIG. 2.—Same Specimen as Fig. 1, Etched in Picral.

(*a*) Surface.

(*b*) Surface Layer Removed.

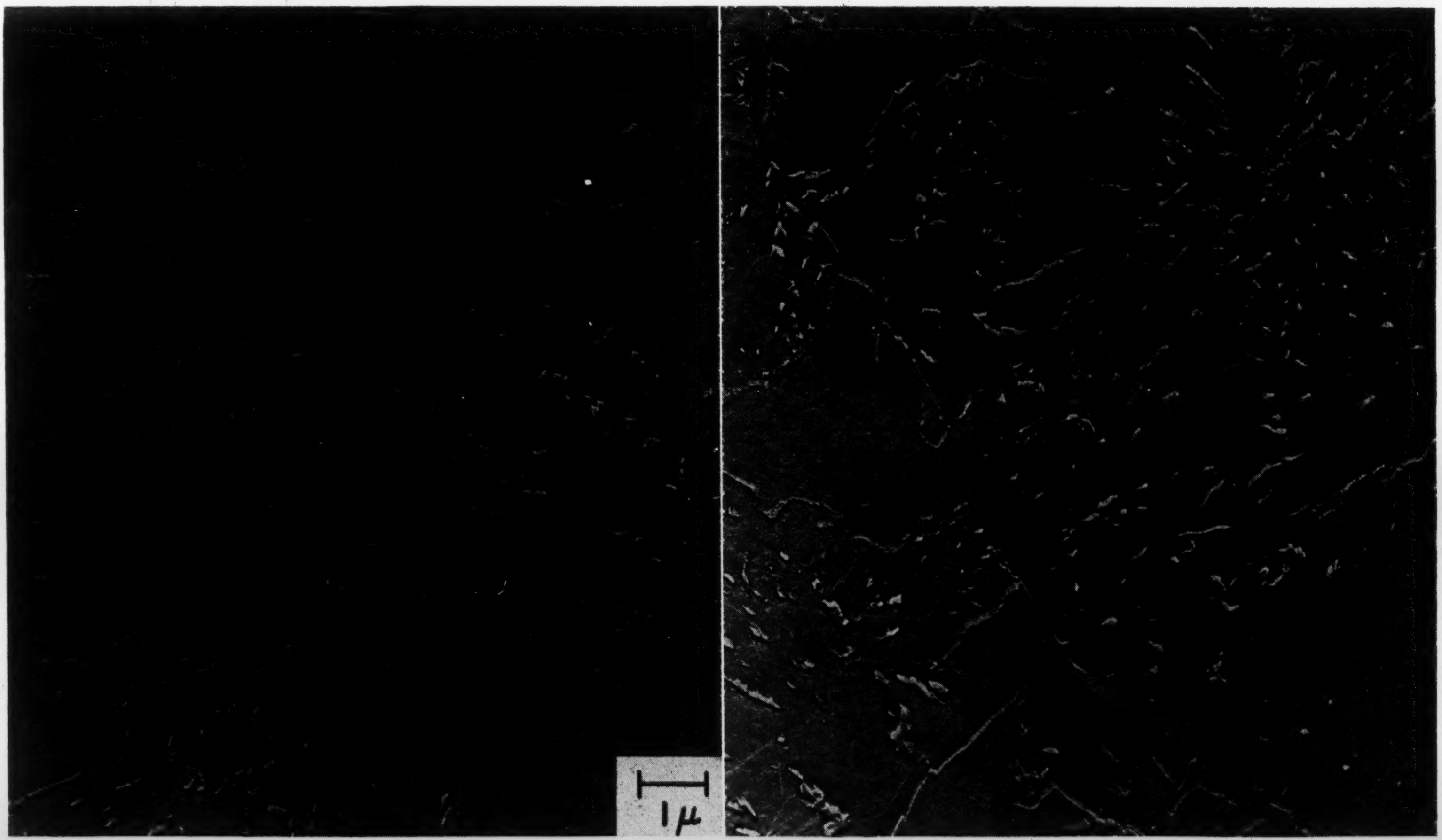

FIG. 3.—Nickel-Chromium Steel; Austenitized at 1600 F, Cooled Quickly to 1100 F; then Cooled at 1 F per sec to 600 F.
(*a*) Surface. (*b*) Surface Layer Removed.

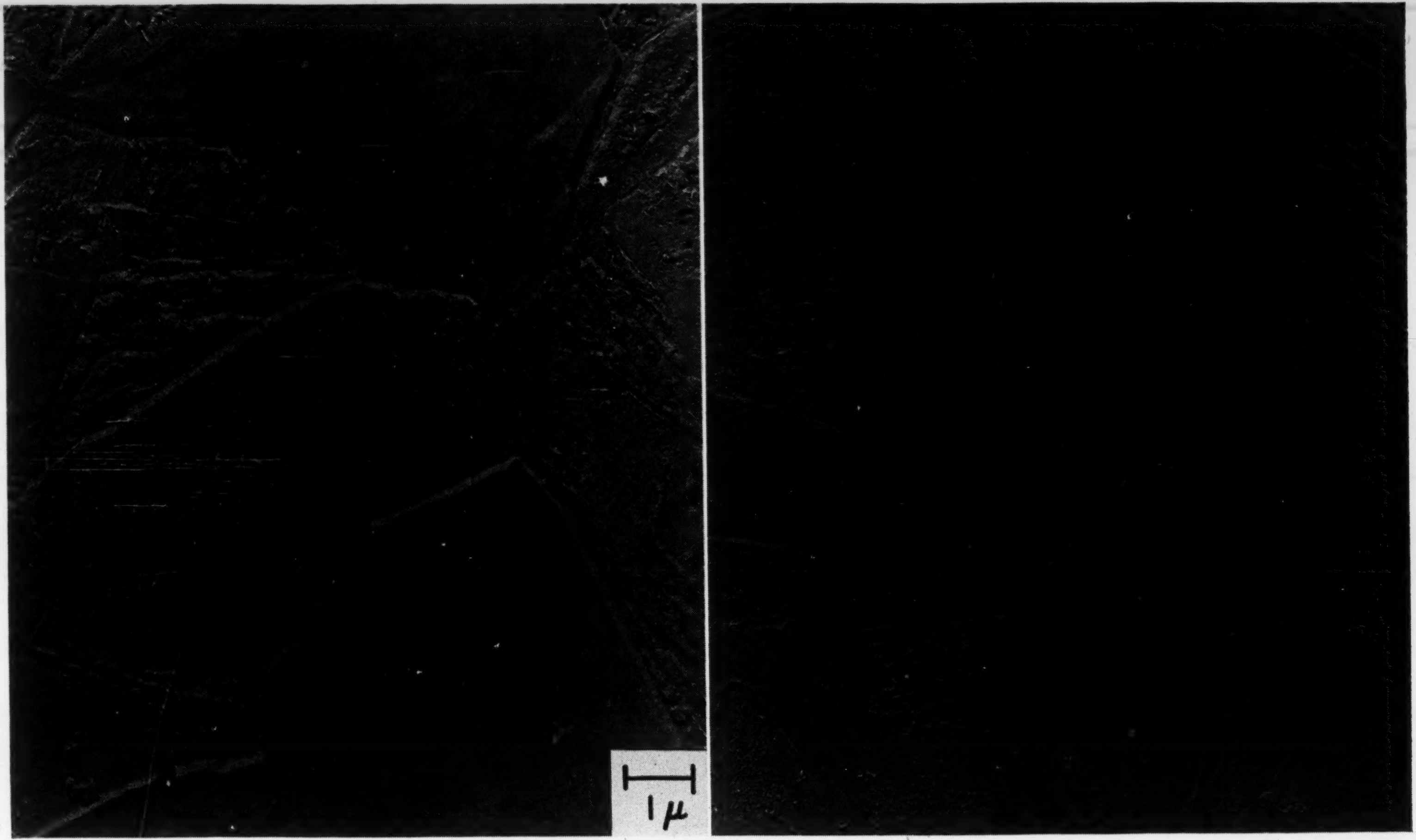

FIG. 4.—Nickel-Chromium Steel; Austenitized at 1600 F; Isothermally Transformed at 1200 F to Form 30 per cent Ferrite, then Quenched to Form Martensite.
(*a*) Surface. (*b*) Surface Layer Removed.

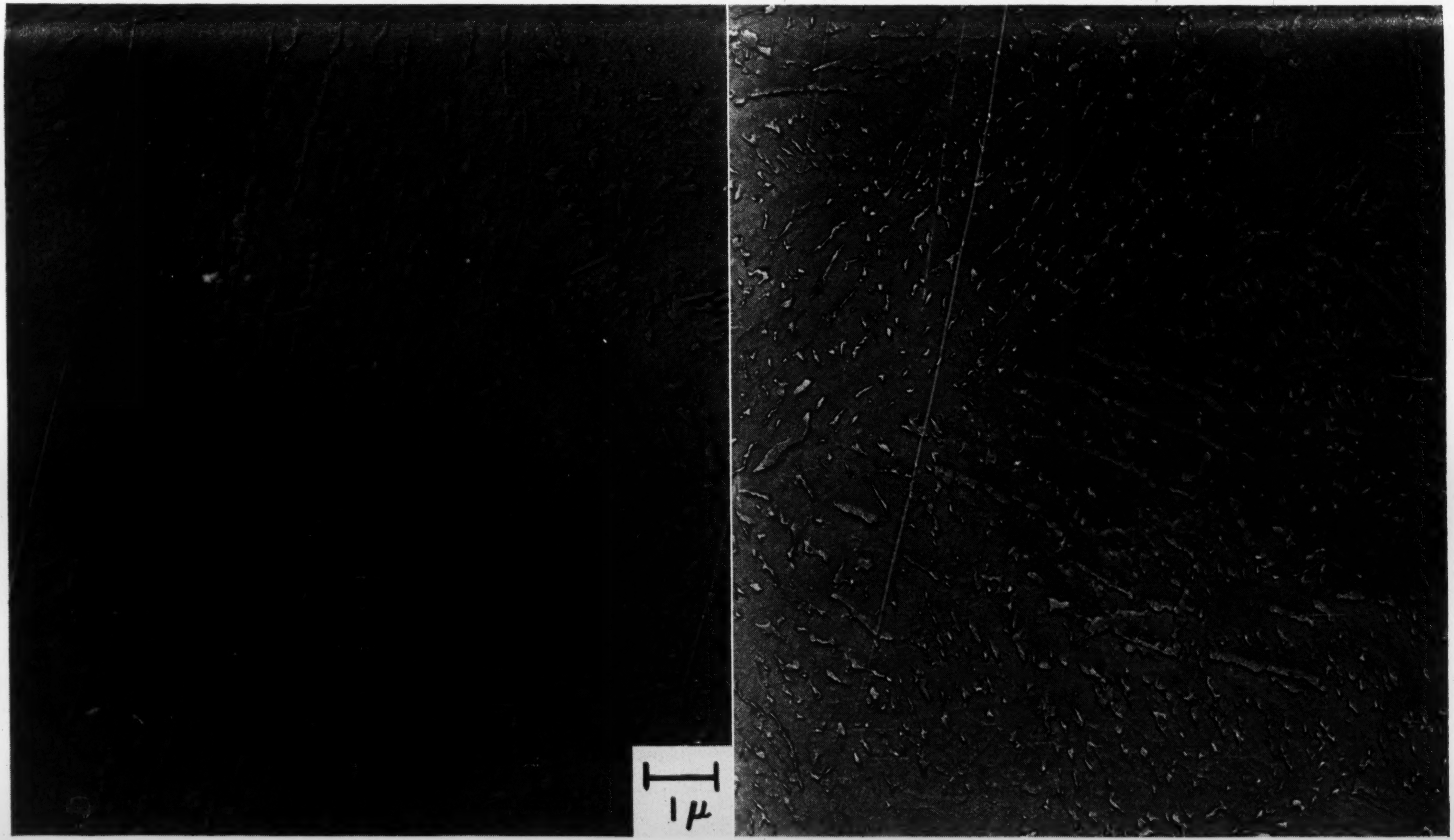

FIG. 5.—AISI 4140 Steel; Austenitized at 1800 F, Cooled Quickly to 800 F, then Cooled at 3 F per sec to 600 F.
(*a*) Surface. (*b*) Surface Layer Removed.

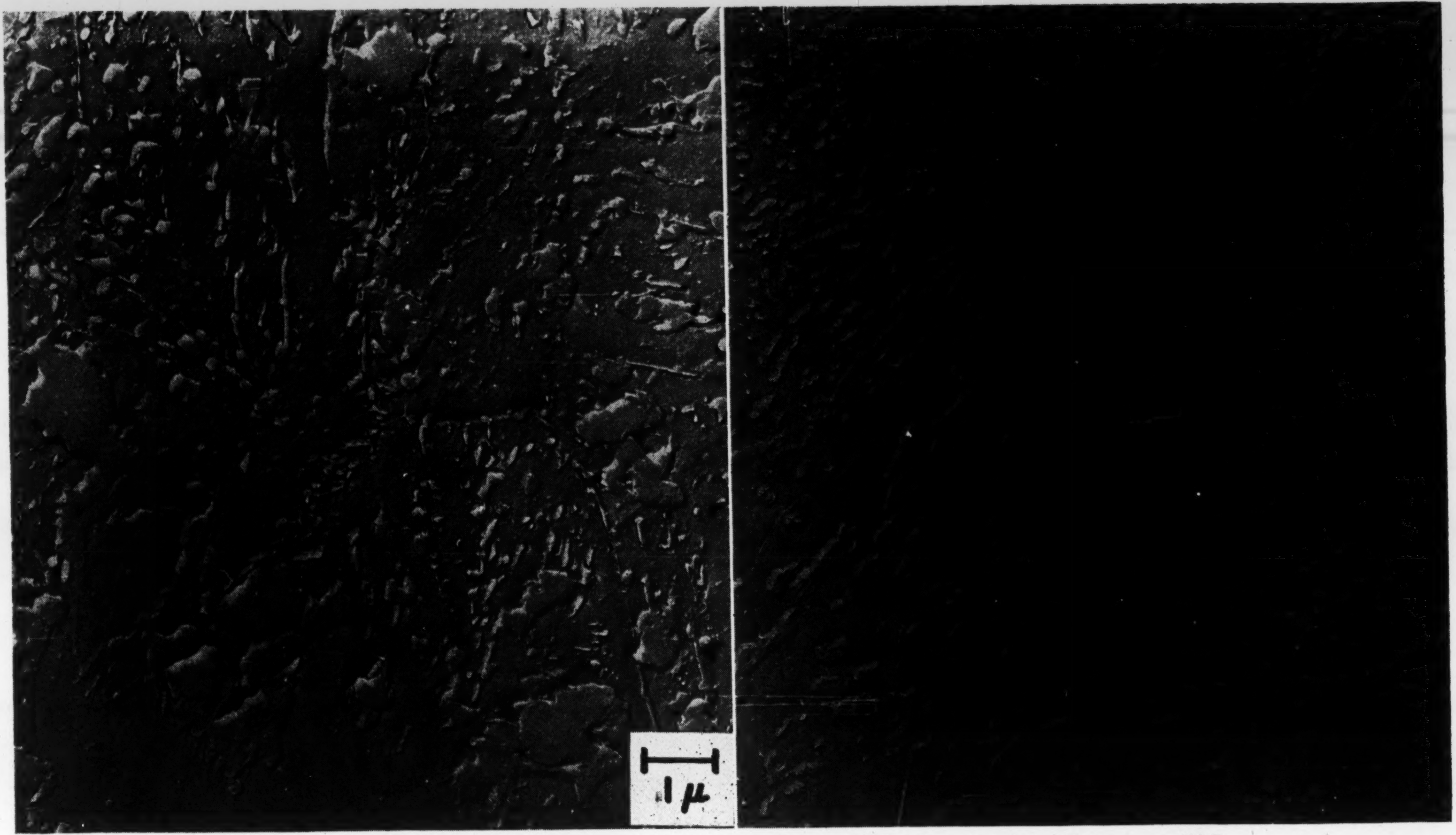

FIG. 6.—AISI 4140 Steel; Austenitized at 1800 F, Cooled Quickly to 900 F, then Cooled at 3 F per sec to 600 F.
(*a*) Surface. (*b*) Surface Layer Removed.

a light chemical etch has been applied to the specimen of Fig. 1. Figure 2 (*b*) is the same specimen again after removal of the surface. From the similar size and distribution of carbides in Figs. 2 (*a*) and (*b*) it is concluded the bainite transformation at the surface is similar to that below the surface. Small patches of martensite are visible in both 2 (*a*) and(*b*).

Figures 3(*a*) and (*b*) represent the surface and volume of the same steel transformed to bainite by continuous cooling at 1 F per sec from 1100 F after austenitizing at 1600 F. Again the appearance of the carbides is the same at the surface and in the volume of the material.

On some areas of the samples of Figs. 2 and 3 the martensite patches had a slightly different appearance at the surface and in the volume of the material. A sample of the same steel was isothermally transformed for 5 min at 1200 F to give about 30 per cent ferrite and then cooled quickly to make the remainder martensite. Figures 4(*a*) and (*b*) show the surface and volume of this specimen: the smooth areas are ferrite. Although the shape and distribution of the martensite areas is about the same, the fine structure within the martensite is different at the surface than in the volume. This is not too surprising since the shear mechanism by which martensite is formed is the most likely mechanism to be affected by the different physical constraint at the surface. No interpretation of the different appearance has been attempted as yet.

4140 Steel:

Similar experiments were made with 4140 steel from the same heat as used by Subcommittee XI for the 5th Progress Report.[3] Figures 5(*a*) and (*b*) show the surface and volume of bainite formed by continuous cooling at 3 F per sec from 800 F after austenitizing at 1800 F. At this rate of cooling most of the bainite was formed between 800 F and 700 F. Not only are the surface and volume similar in appearance, but the size and distribution of the carbides is very much like that in the committee specimen No. 49 isothermally transformed at 700 F. Figs 6(*a*) and (*b*) show the surface and volume of a specimen transformed by continuous cooling at 3 F per sec from 900 F. Most of the bainite was formed between 900 and 800 F. Again the surface and volume are similar and very much like the committee sample No. 47 isothermally transformed at 900 F.

CONCLUSIONS

Several conclusions may be drawn from the results shown above. First, for both the AISI 4140 and the Nickel-Chromium alloys, the bainite at the surface and in the volume of the material was indistinguishable. Furthermore, it seemed unimportant whether it was formed isothermally or by continuous cooling as long as transformations in the same temperature region were compared. Second, there seems to be some difference in the martensite on the surface and in the volume of the material, but the difference is not easily interpreted.

At least in the bainite region, it appears that results from high-temperature X-ray diffraction and from thermal emission electron microscopy (both of which utilize the free surface of a specimen) can be interpreted without error.

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[3] Fifth Progress Report of Subcommittee XI of ASTM Committee E-4. *Proceedings.*, Am. Soc. Testing Mats., Vol. 57 (1957).

APPENDIX VI

CHEMICAL POLISHING OF STEEL FOR ELECTRON METALLOGRAPHY*

By Ronald L. Scott[1]

The search for better and simpler methods of surface preparation of metals to be replicated for electron metallography is always continuing. The conventional mechanical polishing techniques are very satisfactory for most steels and a good deal of work has been done, both independently and cooperatively (**1, 2, 3, 4**),[2] using mechanical polishing methods. However, at various times the question has arisen as to whether mechanical polishing cold works the surface metal and underlying layers sufficiently to cause microstructural changes, particularly in low-temperature isothermal transformation structures and in as-quenched and low-temperature tempered structures. Some of the difficulties encountered by members of Subcommittee XI in their study of low-temperature tempered martensite tend to confirm this suspicion. The work of Beu and Koistinen (**5**) on the effects of metal removal also indicate that microstructural changes can be induced by mechanical polishing.

Preliminary Considerations

A number of solutions are known to give satisfactory results by electrolytic polishing. However, some of these solutions could be dangerous if improperly handled. In addition, a somewhat elaborate apparatus is required to obtain optimum results and the residual etch left on the specimen after electropolishing was often too deep. It was, therefore, decided that chemical polishing should be investigated because of its relative safety and simplicity. It remained to be shown, however, that the residual etches left after polishing would not be too deep for electron metallography.

An aqueous solution of oxalic acid, hydrogen peroxide, and sulfuric acid was suggested by Marshall (**6, 7**) in 1951 for chemical polishing of steels. He was concerned largely, however, with applications of this process to commercial smoothing operations and for surface preparation prior to electrodeposition. A report on the use of this solution for light metallography and production quality control has been made by Sachs and Bunton (**8**) in England.

A modification of Marshall's solution which excludes the sulfuric acid was recommended by Graham, Cranston, and Axon (**9**) for light metallography. This was the basic solution used in the present work and had the following composition:

Oxalic acid (100 g per liter)	7 parts by volume
Hydrogen peroxide (3 per cent)	10 parts by volume
Distilled water	11 parts by volume

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* Presented at a meeting of Subcommittee XI on Electron Microstructure of ASTM Committee E-4 on Metallography, Atlantic City, N. J., June 19, 1956.

[1] Research Staff, General Motors Corp., Detroit, Mich.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 457.

To evaluate the effectiveness of the solution for preparing metal surfaces for electron metallography and to develop confidence in our ability to interpret the micrographs, specimens of SAE 1087 and SAE 4140 steels of well-known structure were chosen to be chemically polished. These specimens have been studied in detail, using mechanical polishing, by Subcommittee XI on Electron Microstructure of Metals of ASTM Committee E-4.

POLISHING AND REPLICA PREPARATION

The chemical polishing sequence is, briefly, as follows. The mounted specimens were polished on a rotary vacuum lap through 0000 emery paper. They were then placed for varying periods of time in the chemical polishing solution. Following this, some of the specimens required an additional etch with picral or nital to develop the structure. The surface was then scrubbed with cotton batting soaked in soap and water, rinsed in alcohol, and air dried. Negative Formvar replicas were made and shadowed with chromium at an angle of 45 deg. The specimen surfaces obtained by chemical polishing were not nearly as smooth as specimens mechanically polished and etched in the conventional manner. This caused considerable difficulty in removing good, continuous, strain-free replicas. Consequently, it became necessary to investigate other methods of replicating chemically polished surfaces, and we have experienced some success with a two-step replica technique in which a thick plastic replica is used for the first step.

EXPERIMENTAL EFFECTS

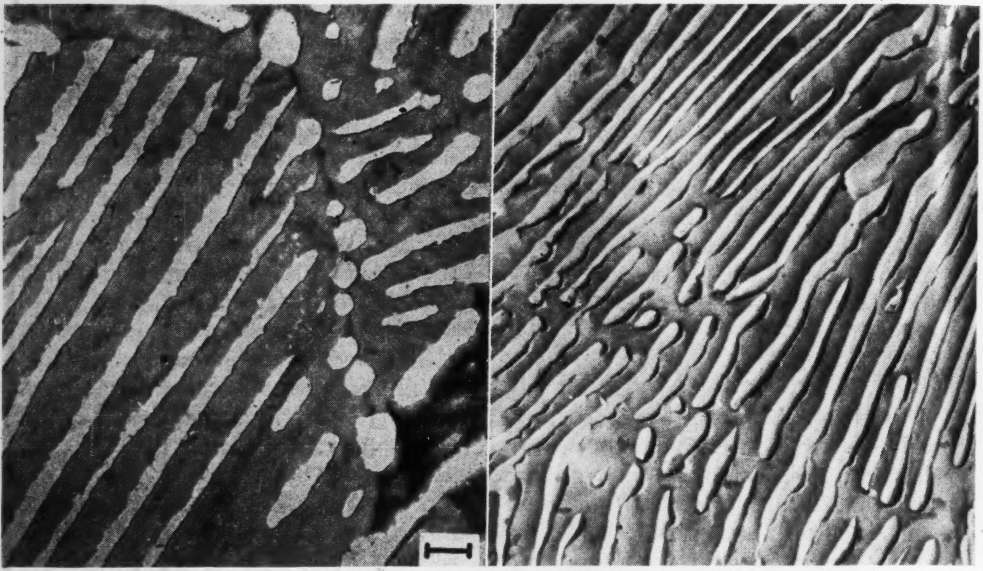
It was observed that the solution did not, in general, impart a surface of high luster (macropolish). Polishing and etching occurred, however, on a microscale such that scratches were gradually reduced in depth with simultaneous development of the microstructure. The softer specimens (high-temperature isothermal transformation and tempered structures) exhibited a satin finish, but the luster increased as the specimen hardness increased.

As might be expected, the reaction rate increased as the temperature was raised. Some of the specimens required elevated bath temperatures (130 F) to reduce polishing time to a more practical value. However, it was frequently noted that pits were formed when the specimen was placed directly in the polishing solution at high temperatures. This difficulty was overcome by starting with the specimen in the bath at room temperature and gradually raising the temperature of the bath to the desired working value.

The effect of changing the ratio of concentration of oxalic acid to hydrogen peroxide was also studied. A high ratio was usually accompanied by a greater dissolution rate with some improvement in the quality of the polish, especially in martensite.

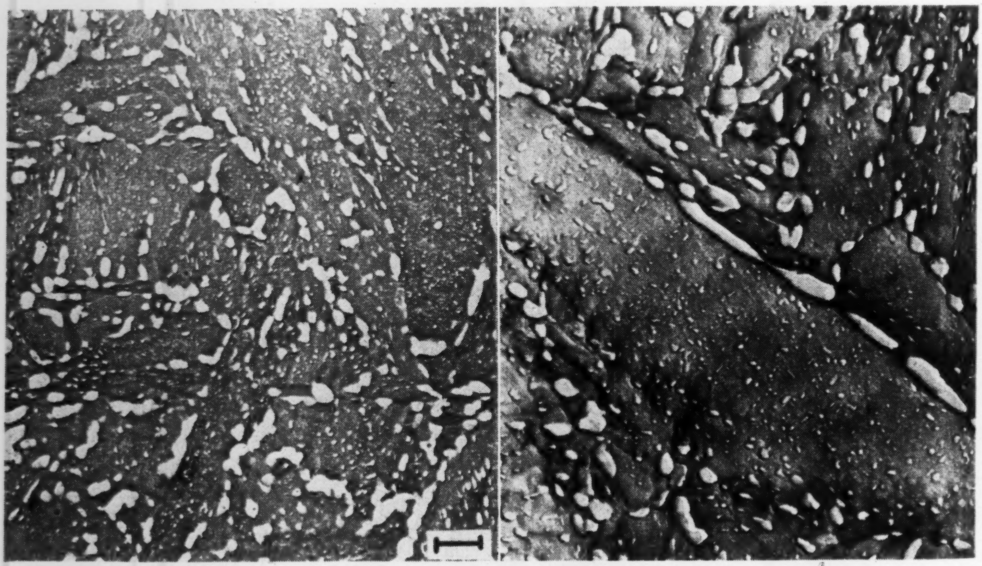
The composition of the steel also has a marked effect upon rate of attack. In general, the SAE 1087 steel was polished more easily than the SAE 4140 steel. In both steels, the high-temperature products, both tempered and isothermally transformed, showed sufficient residual etch after polishing such that no further etching in the usual manner was necessary. On the other hand, the low-temperature isothermal transformation products and the low-temperature tempered specimens required an additional etch after chemical polishing to develop the structure.

One observation of particular interest was made during this investigation. Each specimen was removed from the polishing solution, washed, dried, and placed under the light microscope to determine extent of polishing and



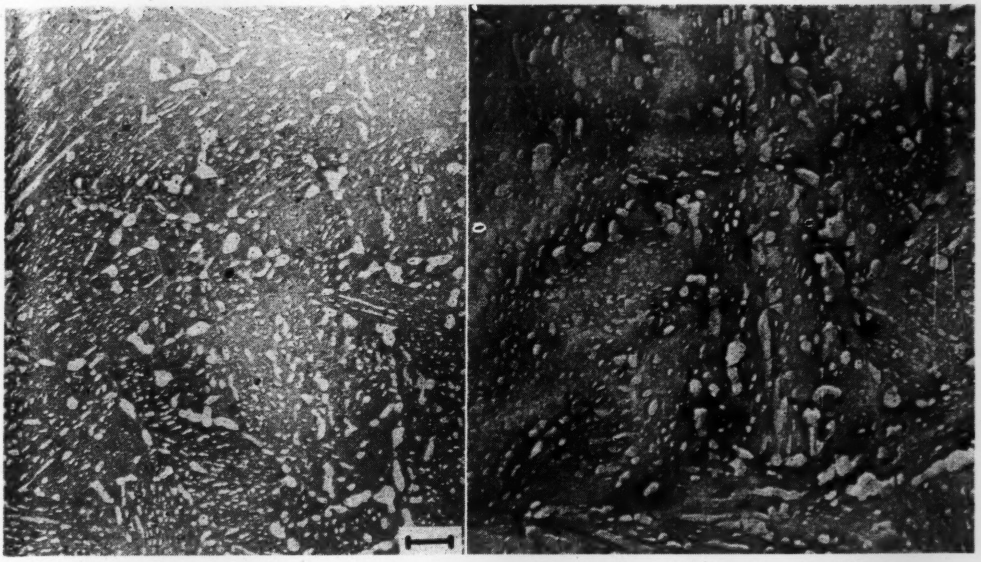
Mechanically polished etch—$FeCl_3HCl$: 30 sec Chemically polished residual etch

FIG. 1.—SAE 1087 Steel—Coarse Pearlite Isothermally Transformed 1300 F—3½ hr.



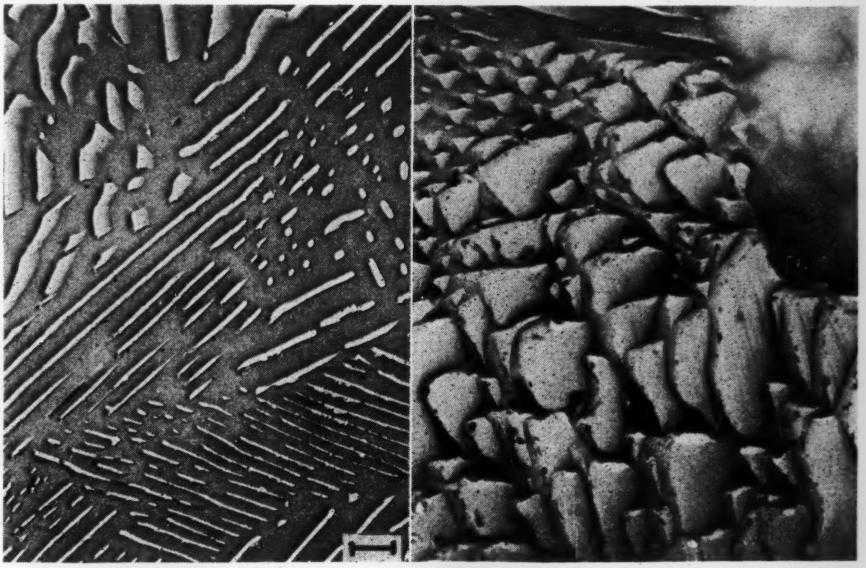
Mechanically polished etch—picral Chemically polished residual etch

FIG. 2.—SAE 1087 Steel—Martensite Quenched and Tempered—1 hr at 1100 F



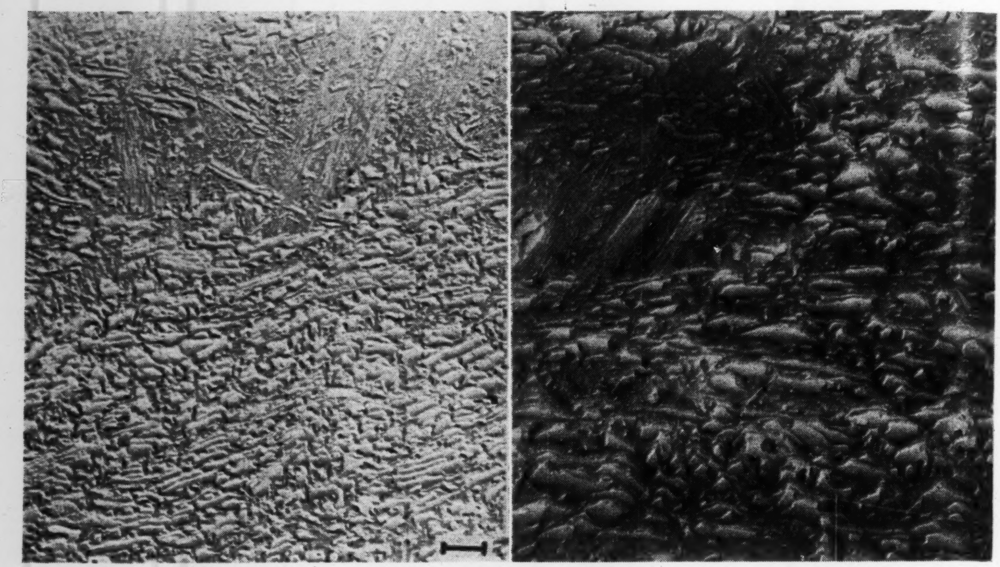
Mechanically polished etch—picral Chemically polished etch—picral 30 sec.

FIG. 3.—SAE 1087 Steel—Bainite Isothermally Transformed 750 F—10 min Tempered 1 hr at 1100 F.



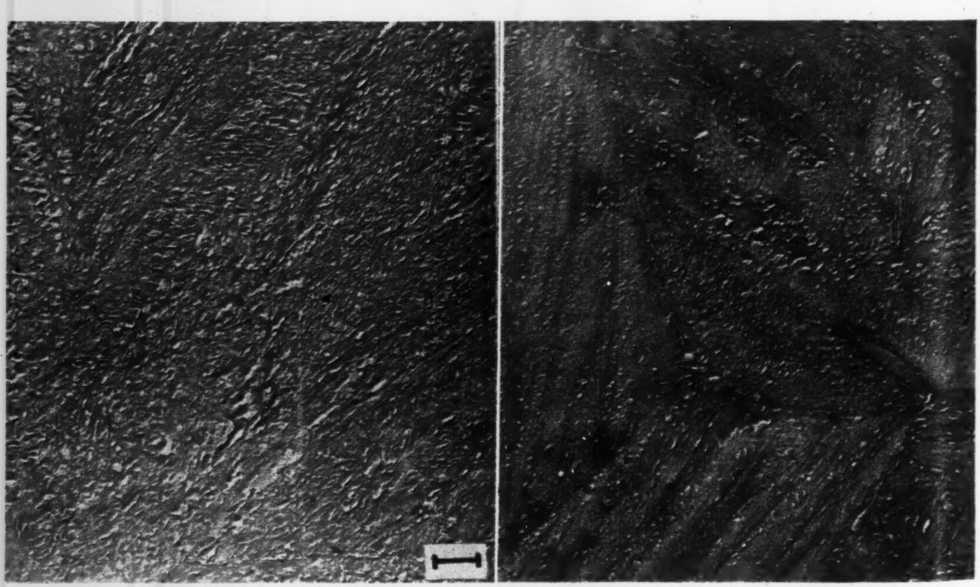
Mechanically polished etch—picral Chemically polished etch—picral 30 sec.

FIG. 4.—SAE 4140 Steel—Pearlite Isothermally Transformed 1300 F—3 hr.



Mechanically polished etch—picral Chemically polished etch—picral 30 sec.

FIG. 5.—SAE 4140 Steel—Pearlite Isothermally Transformed 1100 F—30 min.



Mechanically polished etch—picral 20 sec. Chemically polished etch—picral 4 min.

FIG. 6.—SAE 1087 Steel—Bainite Isothermally Transformed 600 F—30 min.

residual etch. On several occasions when the specimen was again placed in the polishing bath, pits developed. The pits formed quickly and were fairly deep, requiring a polishing time of an hour to an hour and a half for removal. Apparently a protective film which forms on the surface of the specimen during polishing, and constitutes a part of the polishing mechanism, was washed off. When the specimen was again placed in the polishing medium, pitting occurred at highly active points on the surface before the film had an opportunity to reform. The pitting did not occur if these specimens remained in the bath for a long, uninterrupted period of time. It then became apparent that the polishing operation had to be continuous for some types of steel.

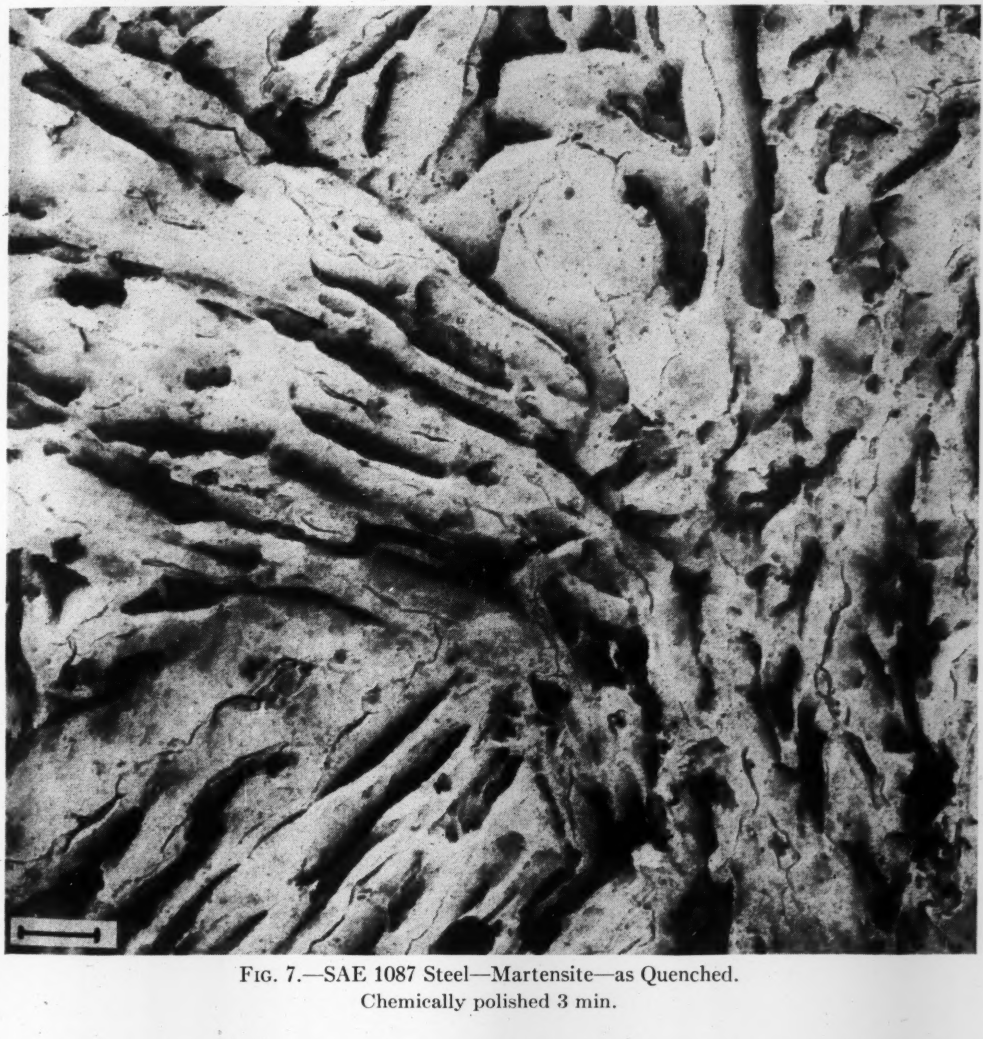


FIG. 7.—SAE 1087 Steel—Martensite—as Quenched.
Chemically polished 3 min.

Although some specimens developed what appeared to be an activated surface in the polishing solution, all of the specimens investigated appeared passive when placed in the usual metallographic etchants. As a result, much longer etching times than usual were required. For example, where 5 to 30 sec was sufficient to etch a mechanically polished surface, up to 4 min was required to etch the same specimen after it had been polished chemically. The reasons for these observed differences in surface reactivity are not understood at this time.

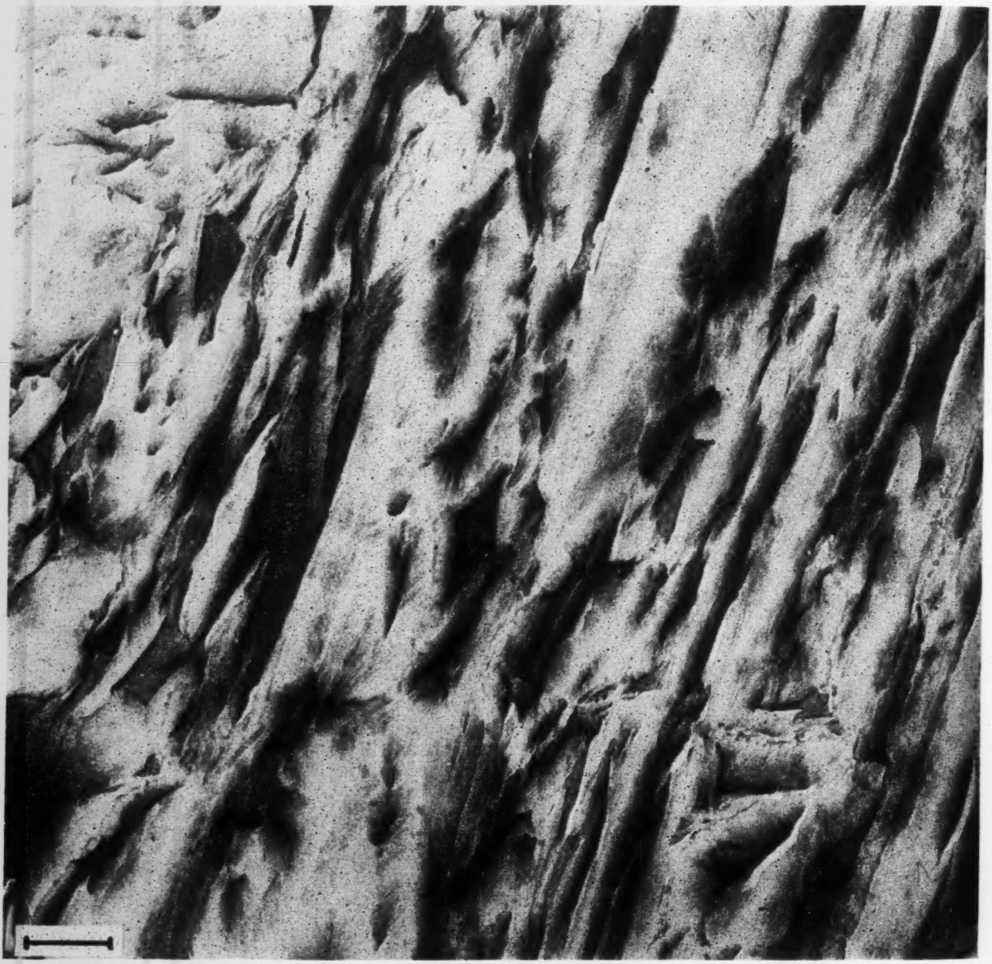


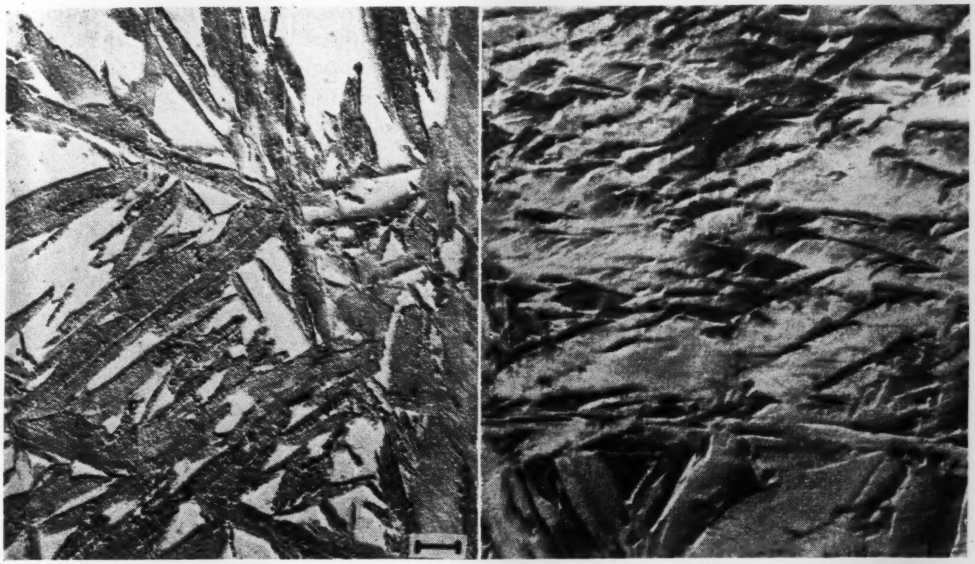
FIG. 8.—SAE 1087 Steel—Martensite—as Quenched. Chemically polished 30 sec.

TEST RESULTS

The electron micrographs shown in Figs. 1 to 14 will demonstrate some of the results that have been obtained on plain carbon and medium alloy steels to date.

In Fig. 1 comparable results on coarse pearlite in SAE 1087 steel are obtained using both methods, except as indicated by the micrographs, the cementite lamellae in the chemically polished specimen have smoother contours.

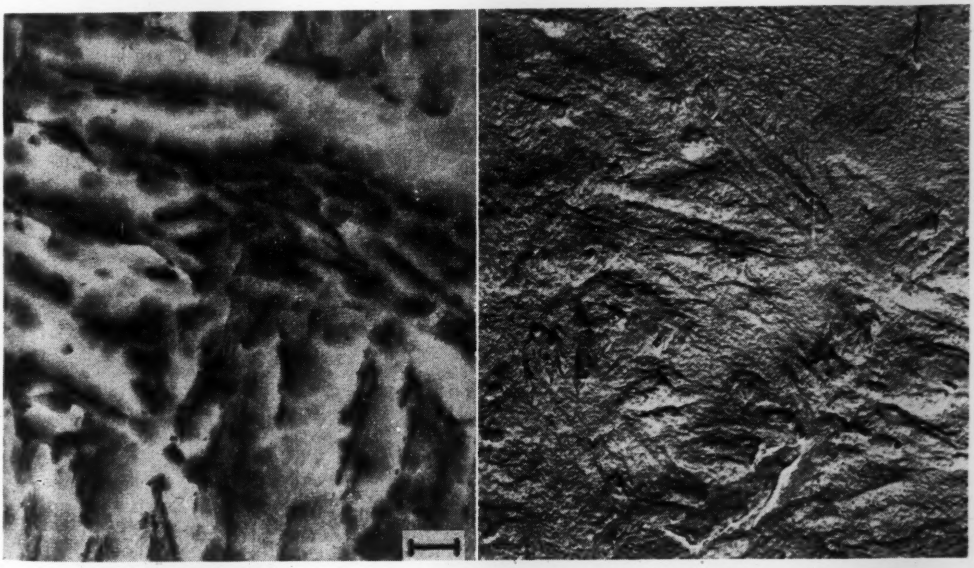
In Fig. 2 similar results are also ob-



Polish—mechanical etch—2 per cent nital + zephiran chloride

Polish—chemical etch—residual

FIG. 9.—SAE 1087 Steel—Martensite—as Quenched.



$\frac{\text{Oxalic Acid}}{H_2O_2} = \frac{7}{10}$

$\frac{\text{Oxalic Acid}}{H_2O_2} = \frac{10}{10}$

FIG. 10.—SAE 1087 Steel—Martensite—Tempered—400 F—1 hr.

tained on the highly tempered structures such as martensite tempered 1 hr at 1100 F. The grain boundary attack, however, is more prominent in the chemically polished specimen and the residual etch is slightly deeper than would normally be used. It appears that it is not too deep, however, to preclude successful replication of this surface.

In Fig. 3 both specimen preparation methods give parallel results on a tempered bainite structure. Here again the residual etch is deeper than that normally used, but satisfactory micrographs can be obtained.

In Fig. 4 the rather dramatic, three dimensional effect observed in the chemically polished specimen here demonstrates well the undercutting of carbides and grain boundary delineation typical

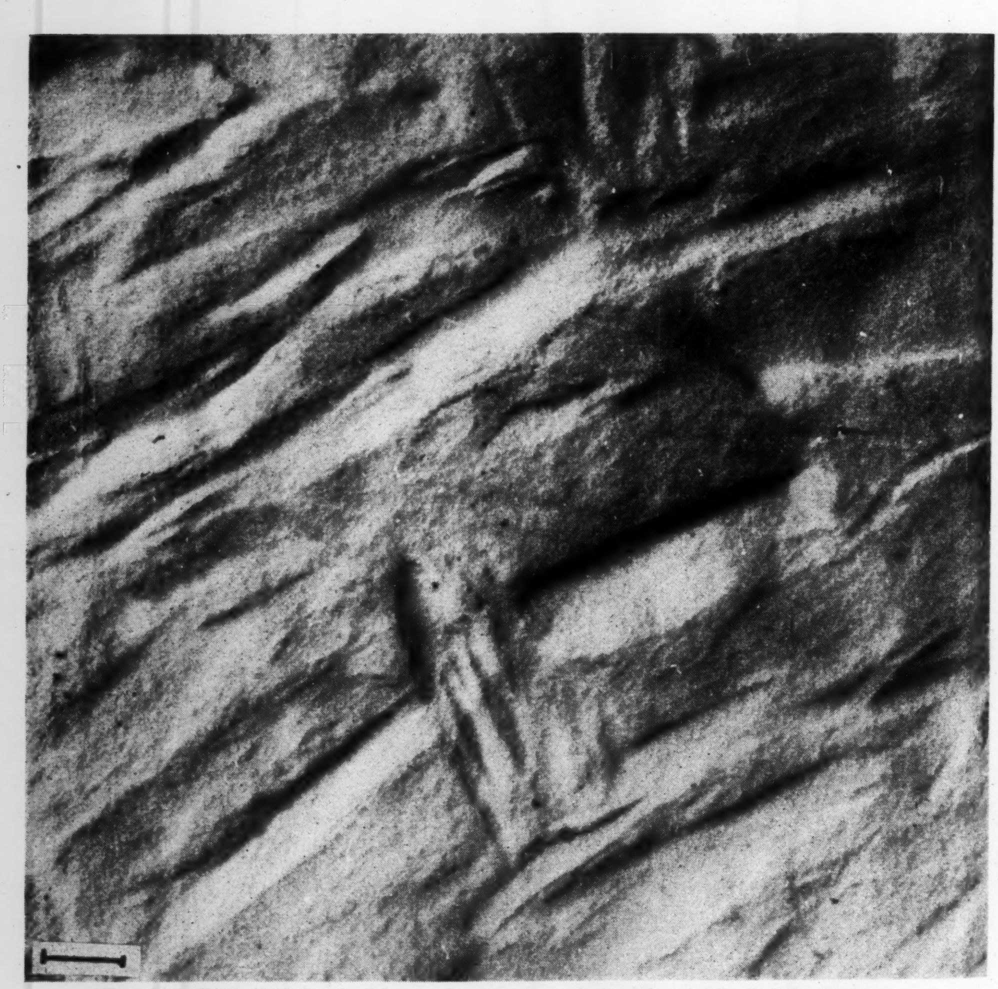


FIG. 11.—SAE 4140 Steel—Martensite—as Quenched—Chemically Polished—60 sec.

$$\frac{\text{Oxalic Acid}}{H_2O_2} = \frac{10}{10}$$

of this polishing solution. The carbides appear to be terraced or laminated. The crystallographic etch apparent in the ferrite is interesting, if not significant. Structures such as this, which tend to tear formvar replicas during stripping, have forced investigators to seek other replicating methods.

In Fig. 5 the two polishing methods have produced almost identical structures in these micrographs of fine pearlite in SAE 4140 steel.

In Fig. 6 the characteristic cross striated lower bainite structure is apparent in both micrographs, though perhaps even more evident in the chemically polished specimen because of grain boundary delineation. The difference in picral etching rates of the two surfaces should be noted.

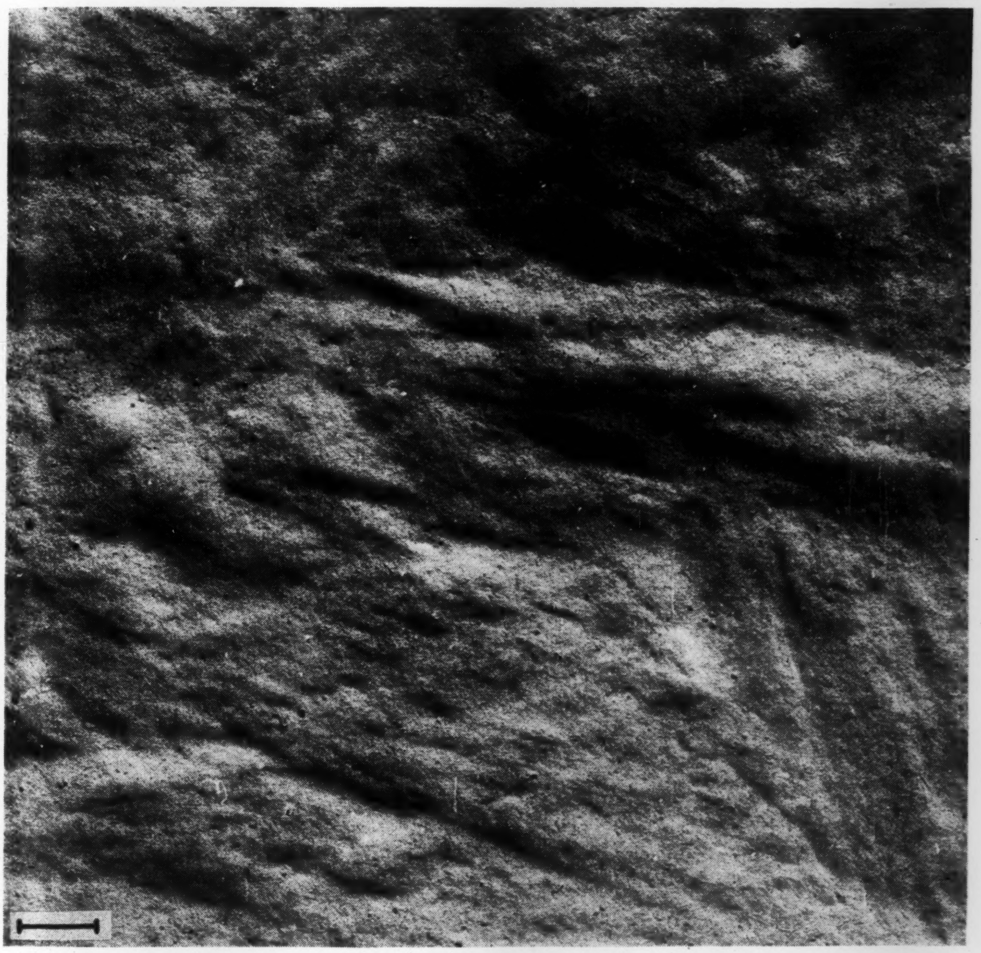


FIG. 12.—SAE 1087 Steel—Martensite—as Quenched—Chemically Polished—45 sec. $\frac{\text{Oxalic Acid}}{H_2O_2} = \frac{10}{10}$

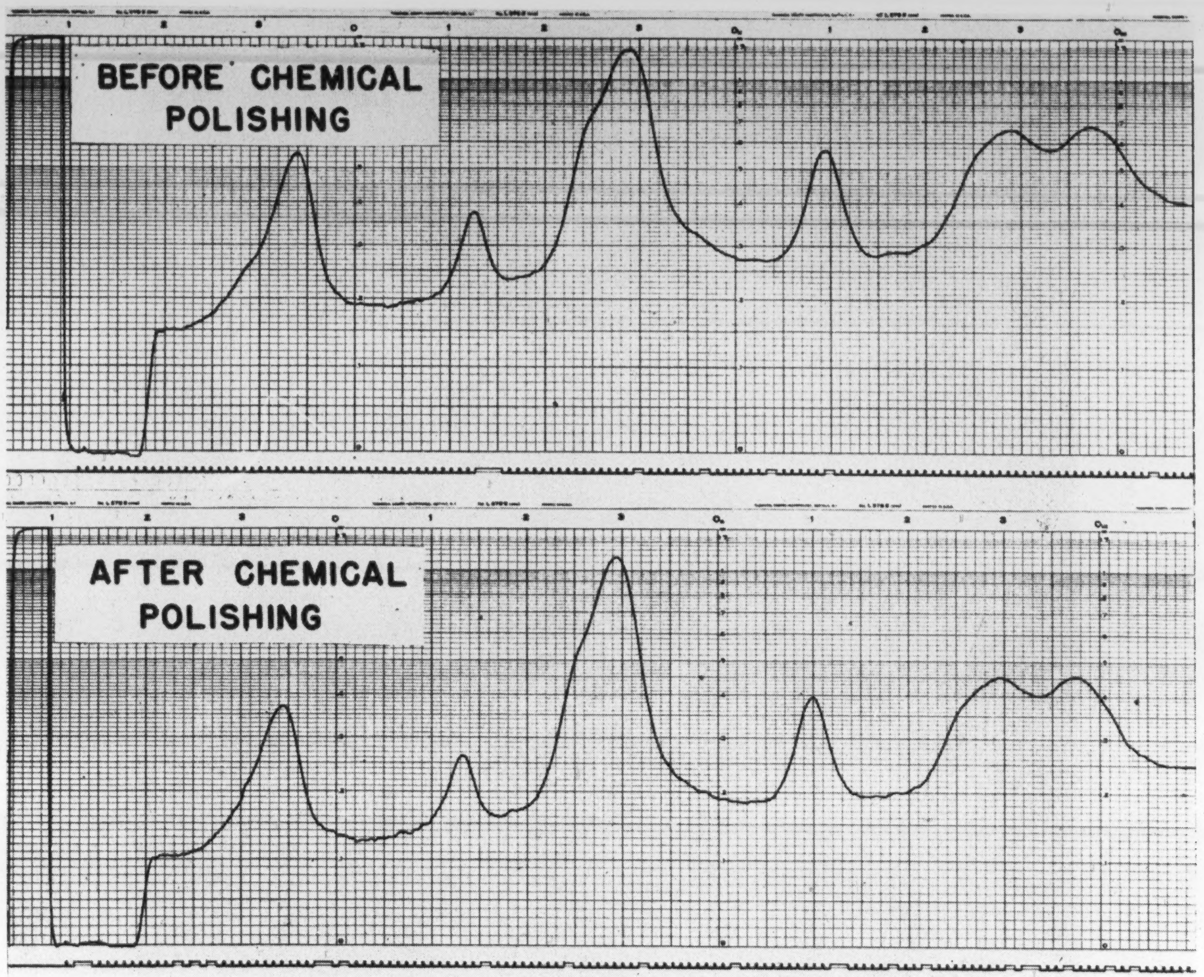


FIG. 13.—X-ray Diffraction Patterns of as Quenched 1087 Steel.

It was mentioned that martensites tempered at 500 F or 600 F were more difficult to polish and etch chemically than those tempered at higher temperatures. It was surprising, however, to find that specimens tempered at 250 F and 350 F and the "as-quenched" specimens were attacked so rapidly that a polishing of two or three minutes was too long, resulting in excessive grain boundary attack (Fig. 7). Polishing for as short a period of time as 30 sec still produced the undesirable grain boundary effect, though it was not so severe (Fig. 8). Then in accordance with the suggestion made by Graham, Cranston, and Axon (9), the oxalate to peroxide ratio was increased and the micrograph on the right in Fig. 9 was obtained.

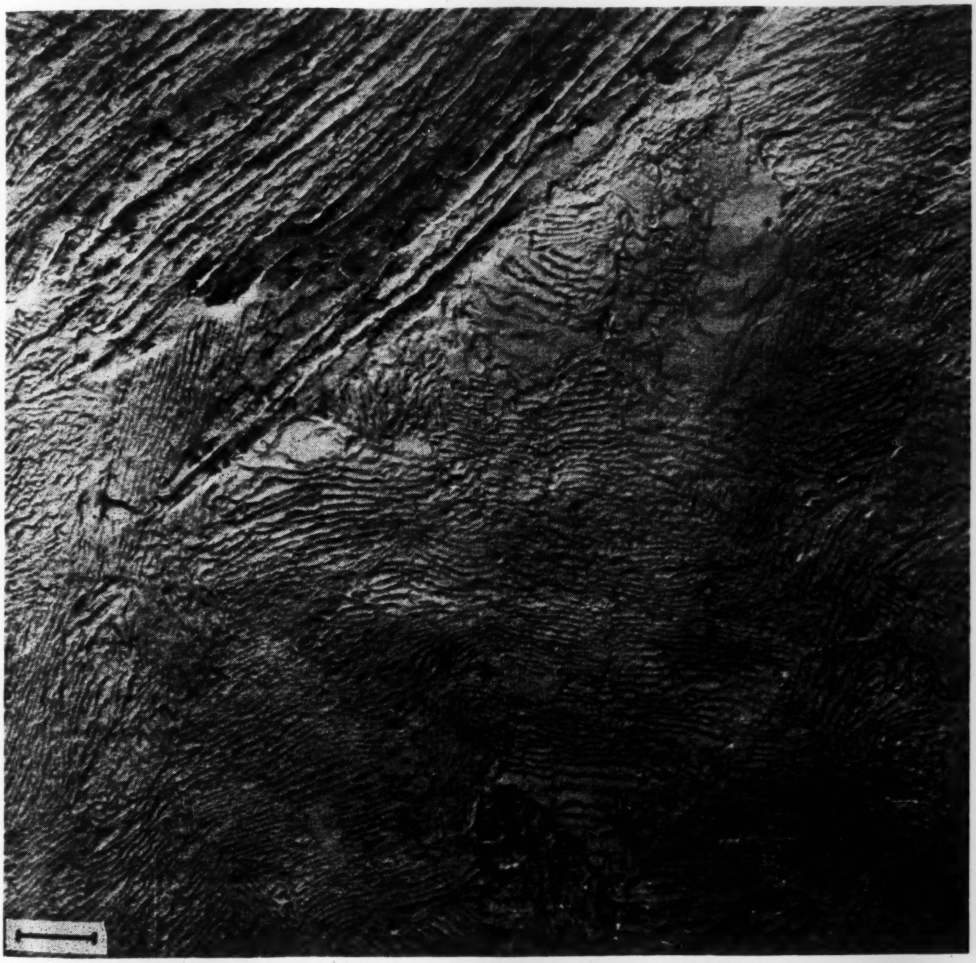


FIG. 14.—SAE 1087 Steel—Fine Pearlite and Bainite—I.T. 6 sec at 550 F—60 sec at 950 F
Chemically polished—3 min.

It can be observed that this specimen presented a reversed surface relief; that is, the retained austenite was etched away by the chemical polishing solution, leaving the martensite in relief. This is in contrast to the effect usually observed when mechanical polishing is used to prepare this steel. During chemical polishing this phenomenon appears to persist in martensites quenched and tempered at temperatures up to the point at which precipitation of epsilon carbide takes place. Similar relief, however, was reported by Ellis (10) using mechanical polishing on high-alloy steel. Further study in this range is needed in order to understand more fully the transformation mechanism.

The excessive grain boundary attack in the left micrograph in Fig. 10 resulted while trying to develop some fine structure which is just barely visible. A change in oxalate-peroxide ratio concurrently with development of the two-stage replica technique resulted in the micrograph on the right.

Figures 11 and 12 show the as-quenched structures of SAE 4140 and SAE 1087 steels as revealed by the higher oxalate-peroxide ratio using the two-stage replica technique. Obviously, the residual etch is insufficient and the next step is to treat these surfaces with nital and zephiran chloride to develop the austenite. It is noteworthy that the martensite in the specimen used for Fig. 12 did not lose its tetragonality during the chemical polishing operation, as shown by the diffraction data shown in Fig. 13. Neither was the retained austenite content decreased.

Figure 14 shows the results of using chemical polishing and a two-stage replica to reveal the structure of upper bainite and fine pearlite. This compares very well with the micrographs of the specimen prepared by mechanical polishing methods.

CONCLUSIONS

The investigations to date have developed the following conclusions:

1. Results thus far indicate that interpretation of electron micrographs of steel polished by both mechanical and chemical methods is similar in most cases.
2. Study of those structures which exhibit reversed relief may add to our present knowledge of the tempering of martensite.
3. In some specimens there appears fine detailed structure which is not found in the same specimen when it has been mechanically polished.
4. The simplified procedure of placing the specimen in a beaker of solution and allowing the polishing action to take place possesses obvious advantages over the mechanical techniques.
5. The non-hazardous nature of the solution and the simplified apparatus required are not to be overlooked as advantages over electropolishing.
6. The polishing solution must be modified for more successful use with some steel specimens. Further, in view of the effectiveness of the present solution, other chemical polishes should be sought for steels and other metals as well.
7. Although it was possible, with persistent effort, to replicate these surfaces using the negative formvar method, to realize in full the more deeply etched surfaces which this method produces, other replica techniques must be investigated.

From the results obtained so far, chemical polishing looks very favorable as a specimen preparation method for electron metallography. It is not proposed, however, as a metallographic panacea though it appears to possess distinct advantages as an adjunct to mechanical polishing. As a tool in electron microscopy, it is still in the early stages of development and it is hoped that further research in this area will be undertaken.

REFERENCES

(1) "Electron Microstructure of Steel," First Progress Report by Subcommittee XI on Electron Microstructure of Steel, ASTM Committee E-4 on Metallography, *Proceedings*, Am. Soc. Testing Mats. Vol. 50, p. 444 (1950).

(2) "Electron Microstructure of Bainite in Steel," Second Progress Report by Subcommittee XI of Committee E-4, *Proceedings*, Am. Soc. Testing Mats., Vol. 52, p. 543 (1952).

(3) "Techniques for Ferrous Electron Metallography," Third Progress Report by Subcommittee XI of Committee E-4, *Proceedings*, Am. Soc. Testing Mats., Vol. 53, p. 505 (1953).

(4) "Electron Microstructure of Tempered Bainite and Tempered Martensite in Steel," Fourth Progress Report by Subcommittee XI of Committee E-4, *Proceedings*, Am. Soc. Testing Mats., Vol. 54, p. 568 (1954).

(5) Karl E. Beu and Donald P. Koistinen, "Some Effects of Metal Rmeoval and Heat Treatment on the Surfaces of Hardened Steels," *Transactions*, Am. Soc. for Metals, Vol. 48, p. 213 (1956).

(6) W. A. Marshall, *Journal*, Electrodep. Tech. Soc., Vol. 28, p. 27 (1952).

(7) W. A. Marshall, *Research*, London, Vol. 7, p. 89 (1954).

(8) K. Sachs and J. D. Bunton, "Non-Destructive Metallography of Plain Carbon Steels," *Metallurgia*, Oct., 1955, p. 205.

(9) L. W. Graham, J. P. Cranston, and H. G. Axon, "Preparation of Steel Specimens for Micro-Examination by a Chemical Polishing Technique," *Research*—Supplement to Vol. 8, No. 4, April, 1955.

(10) A. L. Ellis, "The Electron Microscope in Metallurgy," *Blast Furnace and Steel Plant*, Vol. 38, June, 1950, p. 681–686.

REPORT OF COMMITTEE E-5

ON

FIRE TESTS OF MATERIALS AND CONSTRUCTION*

Committee E-5 on Fire Tests of Materials and Construction held two meetings during the year: on June 28, 1955, in Atlantic City, N.J. and on March 1, 1956 in Buffalo, N. Y.

The committee consists of 58 members, of whom 26 are classified as producers, 6 as consumers, and 26 as general interest members, with 4 consulting members.

The election of officers for the ensuing term of two years resulted in the election of the following:

Chairman, W. J. Krefeld.
Vice-Chairman, R. C. Corson.
Secretary, H. D. Foster.

A draft of a fire test method using small size specimens, prepared by Committee E-6 on Methods of Testing Building Constructions, was considered at the March 1956 meeting. The committee voted to refrain from recommending or publishing the proposed small scale test method principally because the results from such a test could be used intentionally or accidentally for establishing fire resistance ratings of materials in place of the Standard Methods of Fire Tests of Building Construction and Materials (E 119 - 55) which is the ASTM test method for this purpose.

TENTATIVE REVISION OF STANDARD

The committee recommends the following tentative revision of the Standard Methods of Fire Tests of Building Construction and Materials **(E 119 - 55)**:[1]

Section 26.—Change from its present form to read as follows: Where the loading required in Section 24 is not feasible, this alternate test procedure may be used to evaluate the protection of steel beams and girders without application of design load, provided that the protection is not required by design to function structurally in resisting applied loads. The condition of acceptance of this alternate test is not applicable to tests made under design load as provided under tests for Floors and Roofs in Sections 23 to 25.

REVISIONS OF TENTATIVES

The committee recommends that the following two tentatives be revised as indicated and continued as tentative:

Tentative Method of Fire Test of Door Assemblies (E 152 - 55 T):[1]

Section 4 (a).—Change the word "glass" in the next to last line to "vision."

Tentative Method of Fire Tests of Roof Coverings (E 108 - 55 T):[1]

Section 4 (c).—Change the third sentence to read as follows:

> The gas supply shall be predetermined by trial with an auxiliary test deck covered with asbestos cement board to develop a luminous flame with a temperature of 1400 ± 50 F for classes A and B, and 1300 ± 50 F for class C tests as determined by a No. 14 gage chromel-alumel wire thermocouple.

The recommendations in this report have been submitted to letter ballot of

the committee, the results of which will be reported at the Annual Meeting.[2]

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 18-22, 1956.

[1] 1955 Book of ASTM Standards, Part 4.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Fire Tests of Materials and Constructions (A. L. Steiner, chairman) is considering the establishment of more specific requirements for conditioning specimens. Requirements in the existing Method of Test for Fire Tests of Building Construction and Materials (E 119 – 55) pertaining to restraint of wall, partition and floor specimens during the fire test are to be studied for the purpose of establishing specific restraint conditions more representative of actual construction in which building elements may be partially or entirely restrained by adjoining members or portions of the building. The need for a method of determining the load failure of load bearing specimens is still under consideration.

Subcommittee III on Fire Tests of Wall Opening Assemblies (H. D. Foster, chairman).—A preliminary draft of a fire test method for windows and glass block has been prepared and is now undergoing revision in accordance with committee deliberations.

Subcommittee V on Nomenclature and Definitions (J. R. Shank, chairman).—A proposed method of test for use in defining the term "noncombustible" as applied to building materials will be submitted to ASTM Headquarters for publication in the ASTM BULLETIN for comment.

This report has been submitted to letter ballot of the committee, which consists of 58 voting members; 48 members returned their ballots, of whom 43 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

A. L. BROWN,
Chairman.

R. C. CORSON,
Secretary.

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[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE E-6
ON
METHODS OF TESTING BUILDING CONSTRUCTION*

Two meetings of the committee were held during the year: on June 29, 1955, in Atlantic City, N. J. during the Annual Meeting of the Society and on February 28, 1956, in Buffalo, N. Y. during ASTM Committee Week.

During the year the committee went through a period of critical review of its organization, membership, and activities. A revision in scope was approved by the Board of Directors to read as follows:

> To formulate methods of test for building (including housing) construction, including elements, connections and assemblies, under actual or simulated service conditions, applicable to the evaluation of such factors as materials, design, construction and fabrication, with special reference to the needs of building code and similar authorities with whom the committee has liaison.

The committee activities will center around methods of tests pertaining to housing structures and will stress close liaison between building code authorities and ASTM in relation to their needs for standard methods of tests for building constructions.

A new project, under the chairmanship of H. E. Plummer, was authorized to prepare test methods for determining the strength of masonry assemblages.

The officers elected for the ensuing term of two years are as follows:

Chairman, R. F. Legget.
Vice-Chairman, Harold Perrine.
Secretary, R. A. Biggs.
Membership Secretary, J. P. Thompson.

Eleven new members were added during the year bringing the committee membership to 67.

In connection with the Second Pacific Area National Meeting of the Society to be held in Los Angeles, Calif., September 16 to 21, 1956, Committee E-6 is sponsoring a Symposium on Full-Scale Tests on House Structures,[1] and, jointly with Committee D-7 on Wood, the committee is sponsoring symposia on Building Design for Seismic and Shock Loading, and Developments in Glued-Laminated and Other Wood Constructions.[2]

Respectfully submitted on behalf of the committee,

R. F. Legget,
Chairman.

Richard A. Biggs,
Secretary.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17-22, 1956.

[1] Issued as separate publication *ASTM STP No. 210.*

[2] Issued as separate publication *ASTM STP No. 209.*

REPORT OF COMMITTEE E-7
ON
NONDESTRUCTIVE TESTING*

Committee E-7 on Nondestructive Testing met at Atlantic City, N. J., on June 28, 1955, in conjunction with the Annual Meeting of the Society. Several task force, subcommittee, and Executive Council meetings have been held subsequently, notably in Buffalo, N. Y., in February, 1956, during ASTM Committee Week. Further meetings are planned during the 1956 Annual Meeting of the Society.

The committee consists of 135 members, plus 20 consulting members.

The officers elected for the ensuing term of two years are as follows:

Chairman, J. H. Bly.
Vice-Chairman, D. T. O'Connor.
Secretary, Alexander Gobus.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1955 Annual Meeting, Committee E-7 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Recommended Practice for:

Ultrasonic Testing by the Resonance Method (E 113 – 55 T). and
Ultrasonic Testing by the Reflection Method Using Pulsed Longitudinal Waves Induced by Direct Contact (E 114 – 55 T).

The recommended practices appear in the 1955 Book of ASTM Standards, Part 2, bearing the designations indicated above.

TENTATIVES CONTINUED WITHOUT REVISION

In accordance with recommendations of the cognizant subcommittees, the committee recommends that the following tentatives be continued without change for one more year:

Industrial Radiographic Terminology for Use in Radiographic Inspection of Castings and Weldments (**E 52 – 49 T**),
Recommended Practice for Radiographic Testing (**E 94 – 52 T**), and
Reference Radiographs for Inspection of Aluminum and Magnesium Castings (**E 98 – 53 T**).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Radiographic Recommended Practice (E. A. Burrill, chairman) was reactivated during the year and assigned to the project of developing a document or publication correlating the wide variations which can occur in radiographic appearance of flaw and other images when exposure or radiation parameters or specimen thickness are varied over wide ranges. Task forces have been appointed and preliminary work is under way to obtain suitable specimens and determine the scope of investigation required.

Subcommittee II on Reference Radiographs (N. A. Kahn, chairman).—Work continues on the expansion of the Tentative Reference Radiographs for Steel Welds (E 99 – 55 T) to include other types of welds in addition to metallic arc welds. A request has been received from Subcommittee XI on Methods of Testing, of Committee A-3 on Cast Iron, for the expansion of Industrial Radiographic Standards for Steel Castings (E 71 – 52), to include additional flaw types peculiar to nodular irons and cast irons in general.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17-22, 1956.

Subcommittee III on Magnetic Particle and Penetrant Inspection (H. Migel, chairman).—A task group of representatives of several societies under the chairmanship of N. A. Kahn is drafting a set of reference photographs of magnetic particle indications on steel castings, using photographs collected by the Steel Founders' Society, for a special Task Force of Subcommittee III on Oil Piping Systems of Sectional Committee B 31 on Code for Pressure Piping. It is planned that the results of the joint task-force effort will be submitted for approval and publication by ASTM before the end of 1956. Another task force is organizing for work on a specification for penetrant inspection.

Subcommittee V on Radiographic Procedure (C. H. Hastings, chairman) is developing a specification for controlling radiographic quality. A new penetrameter design has been considered, and drafting of an initial document incorporating this penetrameter or a variation of it is under way.

Subcommittee VI on Ultrasonic Testing (J. C. Smack, chairman) prepared the two new recommended practices referred to earlier in the report which were accepted by the Administrative Committee on Standards. Reference Blocks for Ultrasonic Testing and a glossary of terms used in ultrasonic testing are in preparation but will probably not become available before 1957. Task groups are considering ultrasonic inspection of ferrous welds, immersed ultrasonic testing, testing by angle projection of ultrasonic beams introduced by surface contact, and qualifications for ultrasonic operators.

This report has been submitted to letter ballot of the committee, which consists of 135 members; 57 members returned ballots, of whom 50 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. H. BLY,
Chairman.

A. GOBUS,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee E-7 presented to the Society through the Administrative Committee on Standards Proposed Tentative Reference Photographs for Magnetic Particle Testing of Ferrous Castings. The reference photographs were accepted by the Standards Committee on September 5, 1956, and they are available as a separate publication, bearing the designation E 125 – 56 T.

REPORT OF COMMITTEE E-9

ON

FATIGUE*

Committee E-9 held two meetings during the year: on June 30, 1955 in, Atlantic City, N. J., in conjunction with the Annual Meeting of the Society; and on February 27, 1956, in Buffalo, N. Y., in conjunction with ASTM Committee Week.

The committee consists of 47 members, plus 8 consulting members and 1 corresponding member. J. O. Almen was elected to honorary membership of the committee.

Committee E-9 members cooperated with regard to two international conferences on fatigue, namely, the International Conference on Fatigue in Flight Structures held at Columbia University, New York City, on January 31 to February 1, 1956; and the International Conference on Fatigue of Metals being held in London, England, September 10 to 14, 1956, and again in part in New York City on November 28 to 30, 1956.

The officers elected for the ensuing term of two years are as follows:

Chairman, R. E. Peterson.
Secretary, O. J. Horger.

SUBCOMMITTEE ACTIVITIES

Subcommittee I on Research (T. J. Dolan, chairman).—A discussion is planned for the purpose of reviewing applications of solid state physics.

Subcommittee II on Papers (F. M. Howell, chairman).—The following papers relative to fatigue are being presented at the 1956 Annual Meeting:

"Fatigue Properties of Comparable Cast and Wrought Steel," by C. W. Briggs, L. J. Ebert, and E. B. Evans.

"Effect of Changing Cyclic Modulus on the Bending Fatigue Strength," by A. A. Blatherwick and B. J. Lazan.

"Fatigue Crack Propagation in Aluminum Alloys," by M. S. Hunter and W. G. Fricke, Jr.

"The Notched Fatigue Properties of Some Titanium Alloys," by M. J. Sinnott, L. Thomassen, and A. W. Demmler.

"Strengths and Failure Characteristics of AMS 5534A (S-816) Alloy in Direct Tensile Fatigue at Elevated Temperatures," by C. A. Hoffman.

"Statistical Analysis of Fatigue Data," by R. Roeloffs and F. Garofalo.

"An Evaluation of Statistical Treatments of Fatigue Data," by M. N. Torrey and G. R. Gohn.

"A Wire-Fatigue Machine for Investigation of the Influence of Complex Stress Histories," by H. T. Corten and G. M. Sinclair.

Two sessions on fatigue are planned for the Second Pacific Area Meeting of the Society to be held on September 16 to 22, 1956, in Los Angeles, Calif., and proposals for eight papers are under consideration.[1]

Subcommittee III on Survey (H. J. Grover, chairman).—During the year the 1954 References on Fatigue were published as separate publication *ASTM STP 9-F.*

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] See Summary of Proceedings of West Coast Area National Meeting, pp. 52 and 53, for information on where these papers are published.

Subcommittee IV on Large Machines and Test Correlation (J. M. Lessells, chairman).—A Symposium on Large Fatigue Testing Machines and Their Results has been tentatively scheduled for 1957.

Subcommittee V on Aircraft Structures (P. Kuhn, chairman) has cooperated in planning the two sessions on fatigue. Scheduled for the Second Pacific Area Meeting, these sessions deal primarily with fatigue of aircraft structures.

Task Group on Statistical Methods (F. B. Stulen, chairman).—Publication of the "Guide to Statistical Methods for Use in Fatigue Testing" has been withheld pending the completion of certain revisions.

This report has been submitted to letter ballot of the committee, which consists of 47 members; 40 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

R. E. PETERSON,
Chairman.

O. J. HORGER,
Secretary.

REPORT OF COMMITTEE E-10

ON

RADIOACTIVE ISOTOPES*

Committee E-10 on Radioactive Isotopes held two meetings during the year: in Atlantic City, N. J., on June 28, 1955, and in Buffalo, N. Y., on February 28, 1956.

The committee has continued its activities in the field of tests and analyses utilizing radioisotopes, and while the majority of the work has not been completed, there has been prepared a paper on the measurement of carbon-hydrogen ratios with beta particles which will be submitted for publication in the ASTM BULLETIN.

The committee was asked to broaden its scope to include the investigation of changes in the properties and constitution of materials as a function to exposure to radiation, and the following new title and scope have now been approved by the Board of Directors:

Title.—Committee E-10 on Radioisotopes and Radiation Effects.

Scope.—To promote the knowledge of the use of radioisotopes in materials testing and the investigation of the changes in the properties and constitution of materials as a function of exposure to radiation by:

1. Aiding and advising the technical committees of the Society in the preparation of methods and development of instruments of testing.
2. Preparing methods of testing not coming within the scope of other technical committees.
3. Standardizing the nomenclature and definitions used in or relating to testing methods or instruments in cooperation with other committees.
4. Performing such other functions as may advance, improve, standardize, and unify the procedures of materials testing in the fields of radioisotopes and radiation effects.

The committee is sponsoring a Symposium on Applications of Radioisotopes[1] at the Second Pacific Area National Meeting in Los Angeles, Calif., in September, 1956.

The officers elected for the ensuing term of two years are as follows:

Honorary Chairman, G. D. Calkins.
Chairman, C. E. Crompton.
Vice-Chairman, C. E. Weber.
Secretary, J. R. Bradford.

This report has been submitted to letter ballot of the committee which consists of 71 members; 39 members returned their ballots, all of whom voted affirmatively.

Respectfully submitted on behalf of the committee,

G. D. CALKINS,
Chairman.

C. E. WEBER,
Secretary.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] Issued as separate publication *ASTM STP No. 215.*

REPORT OF COMMITTEE E-11

ON

QUALITY CONTROL OF MATERIALS*

Committee E-11 on Quality Control of Materials held two meetings during the year: on November 29, 1955, and on May 22, 1956. Both meetings were held at ASTM Headquarters, Philadelphia, Pa.

The committee suffered loss through the resignation of E. L. Grant of Stanford University. Professor Grant had been a member of the committee since 1953. Four new members have been appointed to the committee: R. A. Bradley, R. J. Hader, G. Lieberman, and C. L. Matz. The committee consists of 22 members. The total allowable membership is 25.

The officers elected for the ensuing term of two years are as follows:

Chairman, H. F. Dodge.
Vice-Chairman, W. R. Pabst, Jr.
Secretary, O. P. Beckwith.

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends that the Tentative Recommended Practice for Probability Sampling of Materials (**E 105 - 54 T**) be continued as tentative since revisions are contemplated therein.

TASK GROUP ACTIVITIES

A survey of sampling plans in ASTM standards, prepared by the Task Group on Sampling Plans, will be submitted for publication in the ASTM BULLETIN. A final draft of a practice for determining the number of tests for a desired precision of an average has been completed and will be sent to the committee for letter ballot. The task group on Design of Experiments has completed a paper on the subject which will be submitted for publication in the ASTM BULLETIN.

This report has been submitted to letter ballot of the committee, which consists of 22 members; 21 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

H. F. DODGE,
Chairman.

O. P. BECKWITH,
Secretary.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee E-11 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Recommended Practice for:

Choice of Sample Size to Estimate the Average Quality of a Lot or Process (E 122 - 56 T)

Revision of Tentative Recommended Practice for:

Probability Sampling of Materials (E 105 - 54 T)

The new Recommended Practice E 122 was accepted by the Standards Committee on August 2, 1956, and appears in the 1956 Supplement to Book of ASTM Standards, Parts 1, 2, 3, 4, 5, 6, and 7; the revision of Tentative Recommended Practice E 105 was accepted on October 29, 1956, and appears in Parts 1, 3, 4, 5 and 6 of the 1956 Supplement.

REPORT OF COMMITTEE E-12

ON

APPEARANCE*

Committee E-12 on Appearance held two meetings during the year: in Atlantic City, N. J., on June 27, 1955, and in New York, N. Y., on April 4, 1956.

The officers elected for the ensuing term of two years are as follows:

Chairman, M. Rea Paul.
Vice-Chairman, George W. Ingle.
Secretary, Richard S. Hunter.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Definitions (R. S. Hunter, acting chairman) has been studying the feasibility of preparing a Manual on Appearance Properties of Materials. Since it does not appear that preparation of such a manual will be possible with volunteer labor, ways are being sought to finance the project.

A group, appointed at the 1955 meeting, has made a survey of all ASTM definitions and has compiled a List of Definitions of Appearance Terms in ASTM Methods as appended hereto.[1]

Subcommittee II on Color (J. A. Van den Akker, chairman) has continued its study of the relationships between visual estimates and measured values of color difference. A project on the color specification of transparent materials is still in the process of organization. A project on total reflectance for acoustical materials and building surfaces is awaiting examples of these materials from other ASTM committees which may be interested in the project.

Subcommittee III on Gloss and Goniophotometry (H. K. Hammond, chairman) is awaiting further interlaboratory comparisons before proceeding with the development of a method of goniophotometry.

Subcommittee IV on Pictorial Representation (R. M. Evans, chairman) has been inactive although there was discussion at the New York meeting of the possibility of enlarging the group's scope to include all visual methods for evaluating appearance quantities.

This report has been submitted to letter ballot of the committee, which consists of 65 members; 35 members returned the ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

M. REA PAUL,
Chairman.

RICHARD S. HUNTER,
Secretary.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] See p. 468.

APPENDIX

DEFINITIONS OF APPEARANCE TERMS IN ASTM METHODS

COLOR

Chromaticity Coordinates, *x*, *y*, *z*.—Tristimulus values expressed as fractions of their total, as follows:

$$x \equiv \frac{X}{X + Y + Z}$$

$$y \equiv \frac{Y}{X + Y + Z}$$

$$z \equiv \frac{Z}{X + Y + Z}$$

(*From Method D 307 – 44.*)

Color.—The aspect of the appearance of objects which depends upon the spectral composition of the light reaching the retina of the eye and upon its temporal and spatial distribution. Black, white, and gray are colors as well as red, yellow, green, blue, purple, and their intermediates. Mixtures or blends of these are also colors. The colors of objects have three attributes: hue, lightness, and saturation.

(*From Definitions D 16 – 52.*)

Color.—The color of an insulating oil is determined by means of transmitted light, and is expressed by a numerical value based on comparison with a series of color standards.

(*From Method D 117 – 54 T.*)

Color Change:

Slight Color Change is one which is perceptible with difficulty.

Appreciable Color Change is one which is readily perceptible without close examination but is insufficient to markedly alter the original color of the specimen.

Extreme Color Change is one which is very obvious and has resulted in a marked alteration of the original color of the specimen.

(*From Methods D 620 – 49 and D 1300 – 53 T.*)

Color Difference between two painted surfaces is the total difference of color, without regard to direction and includes the three attributes—hue, lightness, and saturation; but not other factors of appearance, such as gloss and texture.

(*From Method D 1260 – 55 T.*)

Color Difference.—The resultant vector of three component differences, lightness-index difference, ΔL, and chromaticity differences, Δa and Δb, and may be expressed thus:

$$\bar{E} = (\Delta L)\bar{L} + (\Delta a)\bar{a} + (\Delta b)\bar{b}$$

The above vector notation indicates that ΔL, Δa, and Δb are magnitudes of differences reacting in the direction of $\bar{L}$, $\bar{a}$, and $\bar{b}$, respectively. The latter are unity. The magnitude of this resultant, E, called the color difference (magnitude), is a scalar quantity and may be computed from the following:

$$E = \sqrt{(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2}$$

The magnitude, E, of course gives no indication of the character of the difference, since it does not indicate the relative quantity and direction of hue, saturation, and lightness differences.

(*From Methods D 1365 – 55 T and D 1260 – 55 T.*)

Color Index:

Daylight transmittance, T_g	3.0 to 95.0
Above 95.0	$0.1 = 90 + 0.1\ T_b$
Below 3.0	$0.1 = \log^{-1}[2 - 7.5(2 - \log H)]$

$H = T_g$ for 0.1 *n*-heptane solution

where:

T_b = blue transmittance (see Transmittance).

(*From ASTM Proceedings, Vol. 49, p. 349.*)

Color, Saybolt, is a number related to the depth of column of material the color of which is compared with specified glass standards. The range of numbers is +30 to −16; the greater the number the lighter the color.

(*From Method D 156 – 53 T.*)

Color Space.—The daylight colors of opaque specimens for the purpose of this method are represented by points in a space formed by three mutually perpendicular unit vectors, $\bar{L}$, $\bar{a}$, and $\bar{b}$ having scales L, a, and b, respectively. These scales were chosen so that color differences in this space correlate with perceived color differences.

(*From Methods D 1365 – 55 T and D 1260 – 55 T.*)

Hue.—The attribute which determines whether the color is red, yellow, green, blue, purple, or the like.

(*From Definitions D 16 – 52.*)

Lightness.—The attribute which permits an object color to be classified as equivalent to some member of the series of grays ranging between black and white.

(*From Definitions D 16 – 52.*)

Saturation.—The attribute of any color possessing a hue which determines the degree of the difference from the gray of the same lightness.

(*From Definitions D 16 – 52.*)

Spectral Luminosity, $\bar{y}_\lambda$.—The function of wavelength which serves to define the Y scale, light-producing capacity or luminous efficiency of radiant energy.

(*From Method D 307 – 44.*)

Tristimulus Values, X, Y, Z.—The evaluations of the color as determined in accordance with this method.

(*From Method D 307 – 44.*)

Tristimulus Values of Standard, X_o, Y_o, Z_o.—The color designations of the standard used in the spectrophotometric measurements of the test specimen.

(*From Method D 307 – 44.*)

Tristimulus Values of a Spectrum of Unit Irradiance per Unit Wavelength, $\bar{x}_\lambda$, $\bar{y}_\lambda$, $\bar{z}_\lambda$.—The functions of wavelength which serve to define the X, Y, and Z scales.

(*From Method D 307 – 44.*)

REFLECTANCE

Brightness.—The 45-deg 0-deg directional reflectance of light equivalent of the CIE z function. It is primarily a measure of freedom from impurities left by incomplete bleaching.

(*From Method D 985 – 50.*)

Reflectance.—An abbreviation used in this method for "luminous directional reflectance from daylight relative to magnesium oxide for 45-deg illumination and perpendicular viewing."

(*From Method C 347 – 54 T.*)

Reflectance, Blue-Light.—Blue-light reflectance is the ratio of the light flux from a specimen illuminated at an angle of 45 deg by CIE standard source A, and viewed perpendicularly by a receptor whose response is equivalent to the Z-function of the CIE standard observer, to the light flux from the standard magnesium oxide layer similarly illuminated and viewed.

(*From Method E 97 – 55.*)

Reflectance, Daylight 45-deg, 0-deg Luminous Directional.—Daylight 45-deg, 0-deg luminous directional reflectance (for brevity called reflectance) is the ratio of the luminous flux from a specimen illuminated at an angle of 45 deg by CIE standard source C (average daylight) and viewed perpendicularly by CIE standard observer, to the luminous flux from a standard magnesium layer similarly illuminated and viewed.

(*From Method E 97 – 55.,*

Reflectance, Luminous.—The ratio of the light reflected by any test specimen to that incident on it.

(*From Methods C 347 – 54 T and D 307 – 44.*)

Reflectance, Luminous Directional.—The brightness of a specimen, when illuminated and viewed in a specified manner divided by the brightness that an ideally diffusing, completely reflecting white surface would have when illuminated and viewed in the same manner.

(*From Definitions D 16 – 52.*)

Reflectance, Luminous Directional.—The luminous reflectance which a perfectly diffusing surface would need to possess in order to appear exactly as bright as the test specimen under the same illuminating and viewing conditions.

(*From Methods D 307 – 44 and C 347 – 54 T.*)

Reflectance, Luminous Directional, of a surface for specified directions of illumination and view is the ratio of the brightness of the surface to the brightness that an ideally diffusing, perfectly white surface would have if illuminated and viewed in the same manner.

(*From Method D 1011 – 52.*)

Reflectance, Luminous Fractional.—The ratio of the luminous flux reflected from, to that incident on, a specimen for specified solid angles.

(*From Method D 523 – 53 T.*)

Reflectance, Luminous Total.—The ratio of light reflected by a specimen to that incident on it.

(*From Definitions D 16 – 52.*)

Reflectance Over Black Backing, R_o.—Reflectance of a coating in optical contact with a backing, the ideal reflectance of which is zero and the actual reflectance of which is less than 0.04 (4 per cent).

(*From Method C 347 – 54 T.*)

Reflectance, Spectral.—The ratio of reflected energy to the spectrally homogeneous energy incident on any specimen.

(*From Method D 307 – 44.*)

Reflectance, Spectral Directional. — The spectral reflectance which an ideal diffusing surface would need to possess in order to yield the same appearance as the test specimen under the same illuminating and viewing conditions.

(*From Method D 307 – 44.*)

Reflectivity, R.—The reflectance of a coating so thick that additional thickness does not change the reflectance.

(*From Method C 347 – 54 T.*)

Reflectivity, Luminous.—The luminous reflectance of a test specimen consisting of a layer of material of sufficient thickness that any increase would fail to change this ratio.

(*From Method D 307 – 44.*)

Reflectivity, Luminous Directional, R_s.—The luminous reflectance which a perfectly diffusing surface would have to possess in order to appear exactly as bright as the test material under the same illuminating and viewing conditions.

$$R_s = \frac{\sum_0^\infty R_\lambda H_\lambda \bar{y}_\lambda \Delta\lambda}{\sum_0^\infty H_\lambda \bar{y}_\lambda \Delta\lambda}$$

(*From Method D 307 – 44.*)

Reflectivity, Spectral.—The spectral reflectance of a test specimen consisting of a layer of material of sufficient thickness that any increase would fail to change this ratio.

(*From Method D 307 – 44.*)

Reflectivity, Spectral Directional, R_λ.—The spectral reflectance which an ideal diffusing surface would need to possess in order to yield the same appearance as the test material under the same illuminating and viewing conditions.

(*From Method D 307 – 44.*)

Transmittance

Transmittance, Blue.—The corrected instrument response for a 20-mm layer of sample expressed in percentage transmittance, relative to that for distilled water, and found by use of the tristimulus blue filter specified in Section A3 of Supplement to this method.

(From ASTM Proceedings, Vol. 49, p. 349.)

Transmittance, Daylight.—The corrected instrument response for a 20-mm layer of sample expressed in percentage transmittance, relative to that for distilled water, and found by use of the tristimulus green filter specified in Section A3 of Supplement to this method.

(From ASTM Proceedings, Vol. 49, p. 349.)

Transmittance, Luminous, T_s.—The ratio of transmitted to incident light. It may be calculated from spectral transmittance, T_λ, and spectral luminosity, $\bar{y}_\lambda$, as follows:

$$T_s = \frac{\sum_{0}^{\infty} T_\lambda H_\lambda \bar{y}_\lambda \Delta\lambda}{\sum_{0}^{\infty} H_\lambda \bar{y}_\lambda \Delta\lambda}$$

(From Methods D 307 – 44 and D 1003 – 52.)

Transmittance, Spectral, T_λ.—The ratio of transmitted energy to the spectrally homogeneous energy incident on any test specimen.

(From Method D 307 – 44.)

Gloss

Gloss.—Gloss is the geometrically selective reflectance of a surface responsible for its shiny or lustrous appearance. Surface reflectance is commonly at a maximum in or near the geometric directions in which a mirror would reflect light.

(From Methods D 1222 – 52 T and D 1223 – 52 T.)

Gloss.—Brightness or luster of a body proceeding from a smooth surface.

(From Nomenclature D 675 – 55 T.)

Gloss.—The shine or luster of a smooth enameled surface.

(From Definitions C 286 – 55 T.)

Gloss (Visual Test).—A convenient method of determining apparent gloss is to hold test panel at an angle of incidence approaching 180 deg to a source of artificial light and to note the comparative sharpness of the definition of the image (for example, the lamp filament).

(From Method D 333 – 40.)

Gloss, Percentage, G:

$$G = 100\,\frac{I_1 - I_2}{I_1}$$

where:

I_1 = photoelectric cell reading at the specular angle (45 deg),

I_2 = photoelectric cell reading at the 60-deg angle (30 deg from perpendicular) (15 deg off specular), and

$\frac{I_1}{I_2}$ = measure of the sharpness of the peak of light distribution.

(From Method D 673 – 44.)

Gloss, Specular.—The luminous fractional reflectance of a specimen at the specular direction.

(From Method D 523 – 53 T.)

Gloss, Specular.—The ratio of reflected to incident light, times 1000, for specified apertures of illumination and reception when the axis of reception coincides with the mirror image of the axis of illumination.

(From Method C 346 – 55 T.)

Luster.—That property of a textile material by virtue of which the latter exhibits differences in intensity of light reflected from within a given area of material when the angles of illumination or viewing are changed.

(From Definitions D 123 – 55.)

Mat (Matt or Matte).—A uniformly smooth surface almost, or entirely, lacking gloss.

(*From Definitions C 286 – 55 T.*)

HAZE

Haze.—The haze of a specimen is that percentage of transmitted light which in passing through the specimen deviates from the incident beam by forward scattering. For the purpose of this method only light flux deviating more than 2.5 deg on the average is considered to be haze.

(*From Method D 1003 – 52.*)

Haze.—Indefinite cloudy appearance within, or on the surface of, a plastic not describable by terms of *Chalking*, *Frosting*, or *Bloom*.

(*From Nomenclature D 675 – 55 T.*)

SPECTRAL

Spectral Characteristics.—The appropriate quantity as a function of wavelength by means of which the test specimen is characterized. Spectral transmittance characterizes a light-transmitting specimen; spectral directional reflectivity characterizes a light-reflecting material; and spectral directional reflectance characterizes a light-reflecting object.

(*From Method D 307 – 44.*)

Spectral Irradiance.—The density of radiant flux incident on an element of surface per unit wavelength.

(*From Method D 307 – 44.*)

HIDING

Contrast Ratio:

$$CR = 100 \frac{R_B}{R_W}$$

where R_B and R_W are the apparent light reflectance relative to magnesium oxide, taken as 100 per cent, having standard black backing with apparent reflectance of 0.5 per cent, and standard white backing with apparent reflectance of 91.5 per cent.

(*From Method D 589 – 44.*)

Contrast Ratio, C_{80}.—Ratio of the reflectance of a coating over black backing to its reflectance over a white backing of reflectance of 0.80 (80 per cent).

NOTE.—Contrast ratio varies with the thickness of coating and is a measure of its opacity.

(*From Method C 347 – 54 T.*)

Hiding Power.—The power of a paint or paint material as used to obscure a surface painted with it.

(*From Definitions D 16 – 52.*)

Hiding Power.—The power of a coating to obscure or hide completely the surface over which it is applied.

(*From Definitions C 286 – 55 T.*)

Opacity.—Power of enamel to reflect diffusely a high percentage of incident light.

(*From Definitions C 286 – 55 T.*)

Scatter, Coefficient of, S.—Rate of increase of reflectance with thickness (weight per unit area) at infinitesimal thickness of enamel over an ideally black backing.

(*From Method C 347 – 54 T.*)

MISCELLANEOUS

Diffusion of Light by Plastics, as covered by ASTM Method D 636, needs a separation of Methods A and B into distinct paragraphs to minimize confusion, and definitions like the following:

Reflective Diffusion (Method A):

$$D_{ra} = \frac{\int_{3^\circ}^{90^\circ} B_\theta \, d\theta}{B_{3^\circ}\,(90^\circ - 3^\circ)} = \frac{\sum_{3^\circ}^{90^\circ}{}_\theta \, B_\theta \Delta\theta}{B_{3^\circ}\,(87^\circ)}, \Delta\theta = 1^\circ$$

Transmissive Diffusion (Method A):

$$D_{ta} = \frac{\int_{90^\circ}^{177^\circ} B_\theta \, d\theta}{B_{177^\circ}\,(177^\circ - 90^\circ)}$$

$$= \frac{\sum_{90^\circ}^{177^\circ}{}_\theta \, B_\theta \Delta\theta}{B_{177^\circ}\,(87^\circ)}, \Delta\theta = 1^\circ$$

Reflective Diffusion (*Method B*):

$$D_{rb} = \frac{\int_{15^\circ}^{90^\circ} B_\theta \, d\theta}{B_{15^\circ}\,(90^\circ - 15^\circ)} = \frac{\sum_{15^\circ}^{90^\circ}{}_{\theta}\, B_\theta \, \Delta\theta}{B_{15^\circ}\,(75^\circ)}, \Delta\theta = 1^\circ$$

Transmissive Diffusion (*Method B*):

$$D_{tb} = \frac{\int_{90^\circ}^{165^\circ} B_\theta \, d\theta}{B_{165^\circ}\,(165^\circ - 90^\circ)}$$

$$= \frac{\sum_{90^\circ}^{165^\circ}{}_{\theta}\, B_\theta \, \Delta\theta}{B_{165^\circ}\,(75^\circ)}, \Delta\theta = 1^\circ$$

(See Method D 636 - 54.)

Optical Distortion.—An apparent distortion of anything viewed through a transparent plastic, caused by the nonuniform optical character of the plastic, and not by its shape.

(*From Nomenclature D 675 - 55 T.*)

Orange Peel.—Uneven surface somewhat resembling an orange peel.

(*From Nomenclature D 675 - 55 T.*)

Visual Grading Procedures, lighter and darker, for grading Rosin appear in ASTM Methods D 509. Spectral transmission data are given in Method D 509 for twelve grades of U. S. Rosin. Colorimetric specifications are also given for all types of U. S. Rosin in terms of x, y, T, λ (dominant wavelength), and p (colorimetric purity). No definitions or instructions for computing x, y, T, λ, and p are given. There is also included a Note on color, which states that color (hue, saturation, and brightness) is made up of three attributes as follows:

Hue = characteristic yellow or red,
Saturation = purity or strength of hue, and
Brightness = amount of light transmitted.

(*See Method D 509 - 55.*)

Waviness, Internal.—An appearance of waviness seen *in* a transparent plastic.

(*From Nomenclature D 675 - 55 T.*)

Waviness, Surface.—Wave-like unevenness, or out-of-plane, in the surface of a plastic.

(*From Nomenclature D 675 - 55 T.*)

REFERENCES

Following is a list of the ASTM Standards in which the above Definitions appear:

		1955 Book of ASTM Standards	
		PART	PAGE
C 286 - 55 T.	Def. of Terms Relating to Porcelain Enamel (Tentative)	3	875
C 346 - 55 T.	Method of Test for 45-deg Specular Gloss of Ceramic Materials (Tentative)	3	862
C 347 - 54 T.	Method of Test for Reflectivity and Coefficient of Scatter of White Porcelain Enamels (Tentative)	3	865
D 16 - 52.	Def. of Terms Relating to Paint, Varnish, Lacquer, and Related Products	4	615
D 117 - 54 T.	Methods of Testing Electrical Insulating Oils (Tentative)	5, 6	58, 683
D 123 - 55.	Def. of Terms Relating to Textile Materials	7	1
D 156 - 53 T.	Method of Test for Saybolt Color of Refined Petroleum Products (Saybolt Chronometer Method) (Tentative)	5	95
D 307 - 44.	Method of Test for Spectral Characteristics and Color of Objects and Materials	4	597
D 333 - 40.	Methods of Testing Nitrocellulose Clear Lacquers and Lacquer Enamels	4	449
D 509 - 55.	Methods of Sampling and Grading Rosin	4	686
D 523 - 53 T.	Method of Test for Specular Gloss (Tentative)	4	608
D 589 - 44.	Method of Test for Opacity of Paper and Paper Products	7	849
D 620 - 49.	Method of Test for Colorfastness of Plastics to Light	6	353
D 636 - 54.	Method of Test for Diffusion of Light by Plastics	6	313
D 673 - 44.	Method of Test for Mar Resistance of Plastics	6	251
D 675 - 55 T.	Nomenclature of Descriptive Terms Pertaining to Plastics (Tentative)	6	440
D 985 - 50.	Method of Test for 45-deg, 0-deg Directional Reflectance for Blue Light (Brightness) of Paper	7	980
D 1003 - 52.	Method of Test for Haze and Luminous Transmittance of Transparent Plastics	6	320
D 1011 - 52.	Method of Test for Night Visibility of Traffic Paints	4	497
D 1222 - 52 T.	Method of Test for Contrast Gloss of Paper at 57.5 deg (Tentative)	7	1042
D 1223 - 52 T.	Method of Test for Specular Gloss of Paper at 75 deg (Tentative)	7	1044
D 1260 - 55 T.	Method of Test for Color Difference Using the Hunter Multipurpose Reflectometer (Tentative)	4	594
D 1300 - 53 T.	Spec. and Methods of Test for Laminated Thermosetting Decorative Sheets (Tentative)	6	119
D 1365 - 55 T.	Method of Test for Color Difference Using the Hunter Color Difference Meter (Tentative)	4	590
E 97 - 55.	Method of Test for 45-deg, 0-deg Directional Reflectance of Opaque Specimens by Filter Photometry	3, 4	1899, 1307

ASTM *Proceedings*, Vol. 49, p. 349 (1949).

REPORT OF COMMITTEE E-14
ON
MASS SPECTROMETRY*

Committee E-14 held its fourth meeting in Cincinnati, Ohio, on May 28 to June 1, 1956. A registration of over 300 gives evidence of a continued growing interest in mass spectrometry. A total of 53 papers was presented during the five days, including five papers which comprised a Symposium on the Analysis of Solids. There were also review papers on selected topics.

The officers and members-at-large elected for the ensuing term of two years are as follows:

Chairman, Wm. Priestley, Jr.
Vice-Chairman, V. H. Dibeler.
Vice-Chairman, R. E. Fox.
Secretary, F. W. McLafferty.
Members-at-Large, L. Friedman, and F. W. Karasek.

Activities of Subcommittees

Subcommittee II on Theoretical and Fundamental Aspects (F. L. Mohler, chairman) has prepared a review of the literature covering the following:

1. Ionization and Appearance Potentials and Their Relation to Molecular Properties.
2. Studies of Radicals and Other Transient Configurations.
3. Isotope Abundances and Precision Mass Measurements.
4. Miscellaneous Topics.

The report includes a bibliography of 139 references.

Subcommittee III on High Molecular Weight (M. J. O'Neal, chairman).—Cooperative work is under way with API Projects 42 and 48 on the synthesis of high molecular weight hydrocarbons.

Five laboratories are cooperating on the compilation of tables of isotope abundance in the high molecular weight range.

Subcommittee IV on Methods, Data, and Nomenclature (H. M. Kelly, chairman) is working on instrument performance specifications that will define operation variables and that can be used in methods of test written by product committees.

Many laboratories have accumulated libraries of uncertified spectra, so-called because of the questionable purity of the material or the limited amount of data recorded. Those laboratories that are interested are cooperating to make these spectra available.

Subcommittee V on New Instruments and Techniques (H. F. Wiley, chairman) has prepared a review of the topic from both published and unpublished information available during the past year.

Subcommittee VII on Solids (L. F. Herzog, chairman) sponsored the Symposium on the Analysis of Solids at the recent meeting of the committee.

Respectfully submitted on behalf of the committee,

M. J. O'Neal,
Chairman.

A. B. Kent,
Secretary.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

REPORT OF COMMITTEE F-1

ON

MATERIALS FOR ELECTRON TUBES AND SEMICONDUCTOR DEVICES*

Committee F-1 on Materials for Electron Tubes and Semiconductor Devices held four meetings during the year: in Atlantic City, N. J., an organization meeting on June 27 and 28, 1955, during the Annual Meeting of the Society; in Washington, D. C., on November 3 and 4, 1955; in New York City on February 16 and 17, 1956; and in Swampscott, Mass., on June 14 and 15, 1956.

A highlight of the Washington meeting was a most informative talk on the "Reliability of Vacuum Tubes" by Ray Knight of Aeronautical Research, Inc.

The present membership of the committee totals 52 members, of whom 21 are classified as producers, 24 as consumers, and 7 as general interest members.

COMMITTEE ORGANIZATION

The organization meeting of Committee F-1 was held during the 1955 Annual Meeting of the Society. The nucleus of this committee had previously operated as Subcommittee VIII of Committee B-4. Organization of this committee as a separate group was considered necessary because of the rapid growth of the electronic industry and the need for a committee devoted entirely to electronics.

The officers elected for the ensuing term of two years are as follows:

Chairman, S. A. Standing.
Vice-Chairman, F. J. Biondi.
Secretary, S. Umbreit.

Subcommittees on the following subjects have been organized with the chairmen indicated:

Title	Chairman
I. Cathode Materials	A. M. Bounds
II. Insulators	C. M. Harman
III. Strip	C. W. Horsting
IV. Wire	D. R. Kerstetter
V. Metallic—Non-Metallic Seals	V. J. DeSantis
VI. Semiconductors	H. C. Theuerer
VII. Luminescent Materials	J. G. Koosman
VIII. Editorial	G. S. Szekely

Recognizing this committee's interest in materials for a specific application and covering materials other than non-ferrous metals, the committee has been established as the first of a group with the prefix letter F and is so listed in the 1955 Year Book. Standards and tentatives under its jurisdiction retain their former numerical designation but carry an F- in place of the former B-designation. They now appear in Part 6 of the ASTM Book of Standards.

In view of this new committee's broad interest in materials, liaison is being effected with certain other committees of the Society.

REVISIONS OF TENTATIVES

The committee recommends that the following tentatives be revised as indicated and continued as tentative:

Tentative Methods of Testing Sleeves and Tubing for Radio-Tube Cathodes (F 128 – 55 T):[1]

Revise as appended hereto.[2]

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] 1955 Book of ASTM Standards, Part 6.

[2] The revised Tentatives appear in the 1956 Supplement to Book of ASTM Standards, Part 6.

Tentative Specification for Molybdenum Wire Under 20 Mils in Diameter (F 289 – 54 T):[1]

Revise as appended hereto.[2]

Tentative Method of Test for Diameter by Weighing of Fine Wire Used in Electronic Devices and Lamps (F 205 – 55 T):[1]

Section 2.—In the third sentence, delete the words "flat wound" so that the sentence will read "The torsion balance shall consist of a spring mounted vertically . . ."

Section 3.—Add a new Paragraph (*d*) as follows:

> (*d*) Calibration of the balance, at zero under no load, or at positive values when loaded to check weights, shall be made by using the average of three consecutive readings, taken under conditions specified in Paragraphs (*b*) or (*c*) whichever is applicable.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Specifications for Round Chromium-Copper Wire for Electronic Devices (**F 268 – 53 T**),[1] Tentative Methods of Test for Sag of Tungsten Wire (**F 269 – 52 T**),[1] and for Relative Thermionic Emissive Properties of Materials Used in Electron Tubes (**F 270 – 52 T**),[1] be approved for reference to letter ballot of the Society for adoption as standard without change.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following be continued as tentative without revision:

Specifications for:

Circular Cross-Section Nickel Cathode Sleeves for Electronic Devices (**F 239 – 49 T**),

Tungsten Wire Under 20 Mils in Diameter (**F 288 – 54 T**), and

Round Wire for Use as Electron-Tube Grid Laterals and Verticals (**F 290 – 54 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Cathode Materials (A. M. Bounds, chairman) is continuing its extensive studies of the properties of nickel cathode materials used in the development and manufacture of electron tubes.

Work completed during the year showed that reduction in normal cobalt content, some time ago suspected of being responsible for inferior performance, has in conclusion been exonerated. It is the intent of the committee to recognize appropriately, through a letter by the chairman of the committee, the work done in preparation of test materials by the International Nickel Co.

There has been considerable work and challenge during the year in revising the Specifications for Circular Cross-Section Nickel Cathode Sleeves for Electronic Devices (F 239 – 49 T), and reviewing of views with respect to certain ASTM grade numbers which have occurred therein.

It has been recognized that certain performance characteristics of cathodes are sufficiently influenced by heater problems to warrant investigation. It is expected that appropriate investigation will begin soon.

Other active projects are concerned with a proposed design for a standard planar diode, and tests and specifications for cathodes used in tubes operating at high voltages and under pulse conditions.

It is expected that certification will soon be issued by the National Bureau of

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Standards on two standards of nickel oxide to be identified as Standards Nos. 671 and 672.

Further work continues in the development of a statistical method of evaluating factory prove-in data of new melts in cathode material.

Subcommittee II on Insulators (C. M. Harman, chairman) is largely concerned with studies of mica pieces for "bridges" commonly used in electron tubes and their relation to performance. Such subject matter as distribution of thickness of mica, before and after stamping, and the stiffness of mica is of current interest.

The broader test program is expected to include breakdown temperature of mica under vacuum, the gases given off, delamination strength, behavior to hard and soft X-ray bombardment, surface profile, electrical conductivity of the surface and body under vacuum tube conditions, and standard diode and triode tests.

Subcommittee III on Strip (C. W. Horsting, chairman) is interested presently in the properties of aluminum-clad and nickel-aluminum-clad steel strip, and nickel-plated and nickel-clad steel strip with a carbonized finish, leading to improved specifications.

Subcommittee IV on Wire (D. R. Kerstetter, chairman).—The main interest of this group is in setting up and maintaining standards for the types of wire used for grid laterals and verticals in electron tube manufacture. It has recently reviewed the Methods of Test for Sag of Tungsten Wire (F 269 - 52 T) and have recommended that it be adopted as standard.

A report has been prepared on a test for proportional limit of grid lateral wires with a view that this limit is a significant characteristic in the physical analysis of grid wire.

There has also been some investigation of existing specifications for inner and outer lead wires for miniature tubes.

Subcommittee V on Metallic - Non-Metallic Seals (V. J. DeSantis, chairman) is newly organized and has planned a program for the coming year. The program includes defining a "hermetic" seal; developing tests for glass-to-metal and ceramic-to-metal terminals and methods for the chemical analysis of ferrous sealing alloys; and the effect of minor and trace elements on the physical and mechanical properties of ferrous sealing alloys. Specifications for dumet wire are also in the program.

Subcommittee VI on Semiconductors (H. C. Theuerer, chairman) is actively developing appropriate methods of test and specifications for germanium dioxide. Test methods for moisture, bulk density, volatile material, and the reduction of the dioxide to metal, are being given consideration. The size of bars, single germanium crystals, and the measurement of resistivity of the metal also have been discussed. The subcommittee is attempting to arrive at agreement on a standard size of bar and single crystal.

Subcommittee VII on Luminescent Materials (J. G. Koosman, chairman) is concentrating on specifications relating to particle size of phosphors normally used in cathode ray tubes and is working toward an agreement on a single instrument which may give both an average particle size and size distribution.

Present methods under consideration largely involve liquid settling techniques. Bulk-density measurements are also under consideration as well as specifications for chemical analyses and impurities. The latter activity involves liaison work with other subcommittees of Committee F-1 and perhaps other committees of the Society.

Subcommittee VIII—Editorial (G. S. Szekely, chairman) is set up to review all specifications and methods of test with each of the subcommittees, and to keep them true to ASTM form and editorially correct. Over the past year the subcommittee has editorially reviewed methods F 128, F 205, and F 300.

This report has been submitted to letter ballot of the committee, which consists of 52 members; 38 members returned their ballot, of whom 36 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

S. A. STANDING,
Chairman.

STANTON UMBREIT,
Secretary.

TECHNICAL PAPERS

With Discussions

IMPACT PROPERTIES OF QUENCHED-AND-TEMPERED ALLOY STEELS*

BY JOHN P. SHEEHAN,[1] AND HARRY SCHWARTZBART[2]

Very few single topics have so much aroused the interest and activity of experimental and theoretical research workers in the fields of metallurgy, mechanics, and physics as has the subject of brittle fracture. Although by no means a new problem at the time of World War II, it was brought into dramatic focus at that time by the failure of large numbers of ships and, since then, of other structures such as storage tanks and pipe lines.

Since practical experience with brittle fracture was concerned principally with constructional mild steel, most of the research activity was likewise centered on mild steel. Heat-treated alloy steels are, however, also capable of fracturing in brittle fashion. To give this phase of the problem the attention it deserved, the Office of Naval Research some years ago undertook the sponsorship at the Armour Research Foundation of a comprehensive program on the brittle fracture of quenched-and-tempered alloy steels. This program has now been concluded and it is the intention of this paper to present those phases of the program which have not yet appeared in the literature. Much of the information has been published and the references are given at the end of this paper (**1** to **7**).[3] To give some indication of the scope of the program at this point we might itemize the major variables investigated: effect of carbon content, alloy grade, phosphorus content, boron, rare earth additions, grain size, homogeneity, and specimen size.

GENERAL DISCUSSION

Steel as a material of construction has mechanical properties that are altered by directional and other factors which need to be considered in effective engineering design. Sometimes these factors may be inferred from knowledge of the composition, methods of fabrication, and heat treatment of the steel. Such knowledge is usually supplemented, at least in the more critical applications, by laboratory or full scale testing. In the interest of economy of effort and material, it is desirable that laboratory testing should be used where possible. This requires an understanding of the significance of the test data.

Laboratory tests should be the simplest that will provide the required information. From the standpoint of the engineer, it would be helpful to be able to infer the mechanical properties from readily available information without the performance of laboratory tests other than chemical analysis, or with but simple determinations such as hardness. To a considerable extent it has been possible to predict from hardness determinations the mechanical properties that will be obtained by the conventional tension test, provided that the orientation of the tension specimen in the steel section and the structural state of the steel are known. With a uniform steel structure such as tempered martensite, predictions can be made with considerable assurance that the tensile properties will fall within rather narrow scatter bands.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] Supervisor, Applied Metallurgy, Armour Research Foundation, Illinois Institute of Technology, Chicago, Ill.

[2] Supervisor, Welding, Armour Research Foundation, Illinois Institute of Technology, Chicago, Ill.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 517.

TABLE I.—CHEMICAL ANALYSES OF STEELS INVESTIGATED.

Heat	Nominal Grade	Carbon, per cent	Manganese, per cent	Phosphorus, per cent	Sulfur, per cent	Silicon, per cent	Nickel, per cent	Chromium, per cent	Molybdenum, per cent
3740	1320	0.24	1.60	0.023	0.027	0.23	...	...	...
I	1340	0.40	1.70	0.018	0.031	0.21	0.04	None	Trace
3779	1380	0.73	1.66	0.023	0.022	0.17	...	...	...
2714	2320	0.18	0.91	0.025	0.027	0.17	3.47	...	...
2722	2330	0.33	0.86	0.025	0.027	0.16	3.50	...	...
O	2340	0.43	0.83	0.020	0.028	0.22	3.38	...	...
2725	2360	0.57	0.91	0.025	0.026	0.16	3.50	...	...
2727	2380	0.75	1.00	0.025	0.025	0.19	3.50	...	...
3745	3120	0.23	0.75	0.022	0.020	0.30	1.26	0.58	...
J	3140	0.38	0.77	0.013	0.031	0.19	1.26	0.64	...
3781	3180	0.72	0.92	0.022	0.028	0.25	1.34	0.63	...
3808-3	4120	0.24	0.89	0.019	0.020	0.30	...	0.95	0.21
3808-6	4120P[a]	0.24	0.89	0.031	0.020	0.30	...	0.95	0.21
K	4140	0.40	0.89	0.016	0.026	0.26	...	0.89	0.20
3729	4140P[a]	0.41	0.87	0.037	0.025	0.22	...	0.96	0.21
3814-3	4180	0.74	0.95	0.023	0.029	0.20	...	1.02	0.20
3814-6	4180P[a]	0.74	0.95	0.034	0.029	0.20	...	1.02	0.20
2921	4320	0.21	0.74	0.020	0.016	0.23	1.53	1.09	0.19
2716	4330	0.30	0.84	0.024	0.021	0.31	1.69	1.10	0.20
M	4340	0.38	0.77	0.020	0.035	0.25	1.65	0.93	0.21
2718	4360	0.57	0.87	0.025	0.019	0.23	1.62	1.08	0.22
2720	4380	0.76	0.91	0.024	0.020	0.23	1.67	1.11	0.21
3751	4620	0.20	0.67	0.015	0.011	0.25	1.85	0.30	0.18
L	4640	0.43	0.69	0.009	0.018	0.30	1.78	0.29	0.20
3792	4680	0.74	0.77	0.012	0.011	0.19	1.81	0.30	0.21
3798-3	5120	0.23	0.85	0.023	0.025	0.28	...	1.00	...
3798-6	5120P[a]	0.23	0.85	0.036	0.025	0.28	...	1.00	...
P	5140	0.38	0.80	0.014	0.034	0.19	...	0.87	...
3816	5140P[a]	0.40	0.81	0.037	0.024	0.16	...	0.89	...
3717	5135P[a]	0.35	0.79	0.037	0.031	0.21	...	0.91	...
3813-3	5180	0.72	0.95	0.023	0.029	0.23	...	1.00	...
3813-6	5180P[a]	0.72	0.95	0.035	0.029	0.23	...	1.00	...
2730	8620	0.20	0.89	0.022	0.018	0.14	0.60	0.68	0.20
2820	8630	0.34	0.77	0.020	0.020	0.12	0.66	0.62	0.22
N	8640	0.45	0.78	0.020	0.011	0.16	0.65	0.61	0.20
2928	8660	0.56	0.81	0.018	0.022	0.34	0.70	0.56	0.25
2817	8680	0.76	0.81	0.020	0.011	0.11	0.67	0.60	0.22

[a] Indicates a high phosphorus heat.

The tension test provides the yield strength values needed for design, or the related tensile strength values sometimes employed; but it gives little information, and that indirect, upon the propensity of ferritic steel to fail suddenly under unfavorable combinations of trixial stress pattern, stress concentration, high rates of loading, and low temperatures. The type of laboratory test most frequently used to evaluate the behavior of steel under these conditions is a notched impact test, of which the V-notch Charpy test is one form. This test imposes stress states and rates of loading that while not fully known are nevertheless reproducible. The breaking temperatures of the specimens also are under control. Data from the test are obtained in terms of absorbed energy and macroscopic appearance of the fracture surfaces of the broken specimens.

TABLE II.—NORMALIZING AND AUSTENITIZING TEMPERATURES, AS-AUSTENITIZED GRAIN SIZE.

Heat	Nominal Grade	Normalizing and Austenitizing Temperature, deg Fahr	Austenitic Grain Size, ASTM Classification Number
3740.........	1320	1650	9
I............	1340	1550	8
3779.........	1380	1450	8
2714.........	2320	1650	6–7
2722.........	2330	1575	7–8
O	2340	1550	8–9
2725.........	2360	1475	7–8
2727.........	2380	1450	7–8
3745.........	3120	1650	...
J............	3140	1550	8–9
3781.........	3180	1450	9
3808-3........	4120	1650	8–9
3808-6........	4120P[a]	1650	8–9
K............	4140	1550	8–9
3729.........	4140P[a]	1550	8–9
3814-3........	4180	1450	8–9
3814-6........	4180P[a]	1450	8–9
2921.........	4320	1650	8–9
2716.........	4330	1575	8–9
M............	4340	1550	8–9
2718.........	4360	1475	7–9
2720.........	4380	1450	8–9
3751.........	4620	1650	8–9
L............	4640	1550	7–8
3792.........	4680	1450	8
3798-3........	5120	1650	7–9
3798-6........	5120P[a]	1650	8
P............	5140	1550	7–8
3816.........	5140P[a]	1550	8
3717.........	5135P[a]	1550	7–9
3813-3........	5180	1450	8–9
3813-6........	5180P[a]	1450	8
2730.........	8620	1650	8–9
2820.........	8630	1575	7–9
N............	8640	1550	8–9
2928.........	8660	1475	8–9
2817.........	8680	1450	8–9

[a] Indicates a high phosphorus heat.

Use of the hardness of the steel to predict its behavior in notched bar impact tests has been far less successful than in the case of the tension test. Obviously this comes about because the hardness determination and the impact test measure different combinations among the properties of the steel. Conditions that influence the notched bar data often do not change the hardness at all. Only when the factors that cause deviations in the notched bar data are known and recognized can hardness be used with other readily available knowledge as a telltale of the impact behavior to be expected.

In a previous paper (4) the basic V-notch Charpy behavior of quenched-and-tempered alloy steels was discussed in detail. The relationship of impact energy and fracture with tempering temperature was shown for a number of commercial grades of alloy steel, and the different types of embrittlement that occur with tempering were defined. In addition, the various criteria of transition temperature, based on energy fracture transition, were also discussed.

In soft steel structures, tempered martensite, as well as others, one popular criterion of transition has been the macroscopic rating of 0 per cent brittle fracture. Sometimes another percentage rating of the brittleness of the fractures has been chosen. In soft steel it has been the general observation that 0 per cent brittle fracture frequently represents a point on the upper half of the pronounced inflection that constitutes the energy transition. Within high-hardness ranges, tempered martensite does not show a macroscopically readable change to 0 per cent brittle fracture. Thus, care must be taken not to push comparisons by this criterion too far into the less-tempered martensites. In the present study, 80 per cent of the maximum energy has been chosen as the criterion for this purpose. In the softer tempered martensites, that is, those tempered at about 1000 F and higher temperatures, 80 per cent of the maximum energy corresponded roughly to the index of the 0 per cent brittle fracture, and so was comparable to the transition that has been used in many of the more recent investigations of impact transition temperatures.

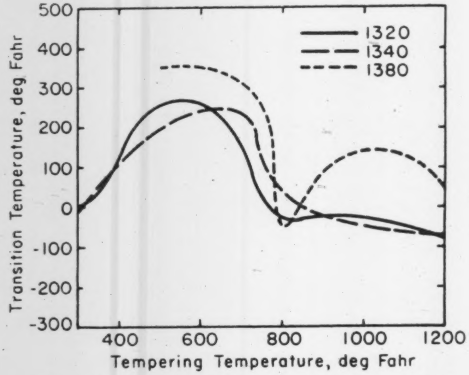

FIG. 1.—Transition Temperature as a Function of Tempering Temperature and Carbon Content for the 1300 Series.

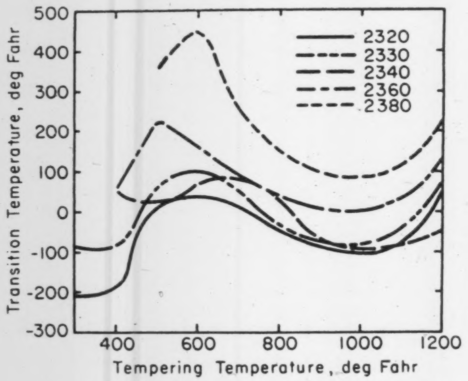

FIG. 2.—Transition Temperature as a Function of Tempering Temperature and Carbon Content for the 2300 Series.

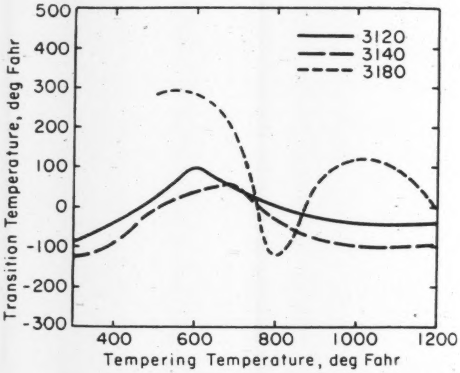

FIG. 3.—Transition Temperature as a Function of Tempering Temperature and Carbon Content for the 3100 Series.

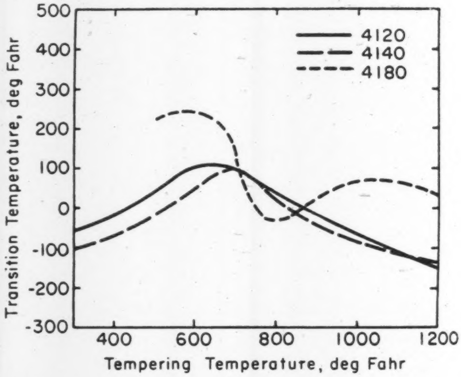

FIG. 4.—Transition Temperature as a Function of Tempering Temperature and Carbon Content for the 4100 Series.

EFFECT OF CARBON AND ALLOY CONTENTS

The following eight grades of alloy steel have been investigated: 1300, 2300, 3100, 4100, 4300, 4600, 5100, and 8600.

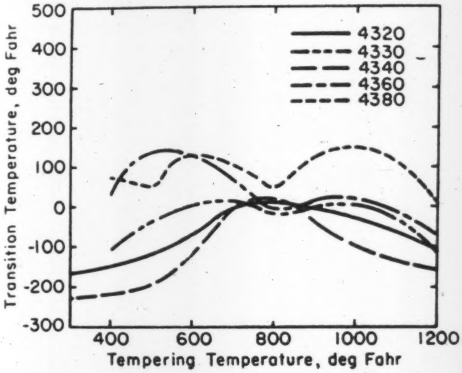

FIG. 5.—Transition Temperature as a Function of Tempering Temperature and Carbon Content for the 4300 Series.

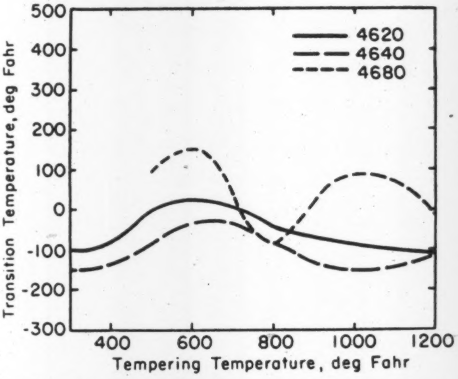

FIG. 6.—Transition Temperature as a Function of Tempering Temperature and Carbon Content for the 4600 Series.

At least three carbon levels, 0.20, 0.40, and 0.80 per cent, were investigated, and for the 2300, 4300, and 8600 series, carbon levels of 0.30 and 0.60 per cent were also tested. The 4100 and 5100 series were investigated at two levels of phosphorus content by utilizing split heats. These series have the same compositions except for a molybdenum content in 4100, but lacking in 5100. Analyses of all the steels investigated are given in Table I.

The heats were made in a 500-lb laboratory induction furnace by fine-grain practice (deoxidized with 1½ lb aluminum per ton). Sixty-five pound ingots were poured from each heat.

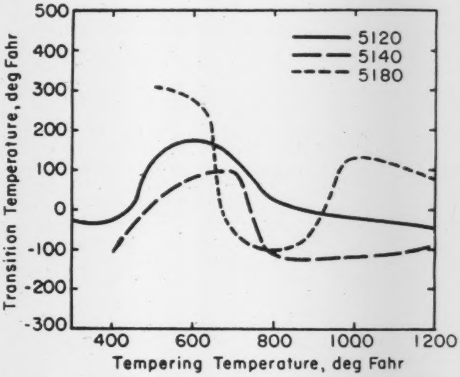

FIG. 7.—Transition Temperature as a Function of Tempering Temperature and Carbon Content for the 5100 Series.

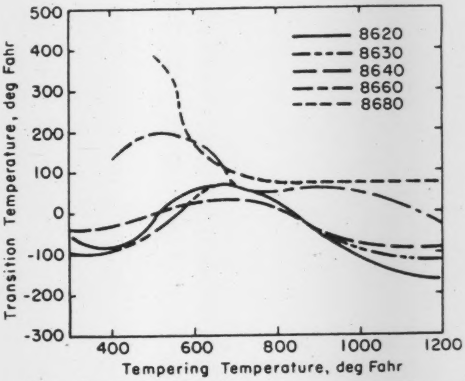

FIG. 8.—Transition Temperature as a Function of Tempering Temperature and Carbon Content for the 8600 Series.

The ingots were forged to ½ by 2½ in. bars from which the impact specimens were cut. To prepare the steel for machining, the forged bars were normalized from the temperatures listed in Table II, then heated for 1 hr at 1200 F, and air cooled.

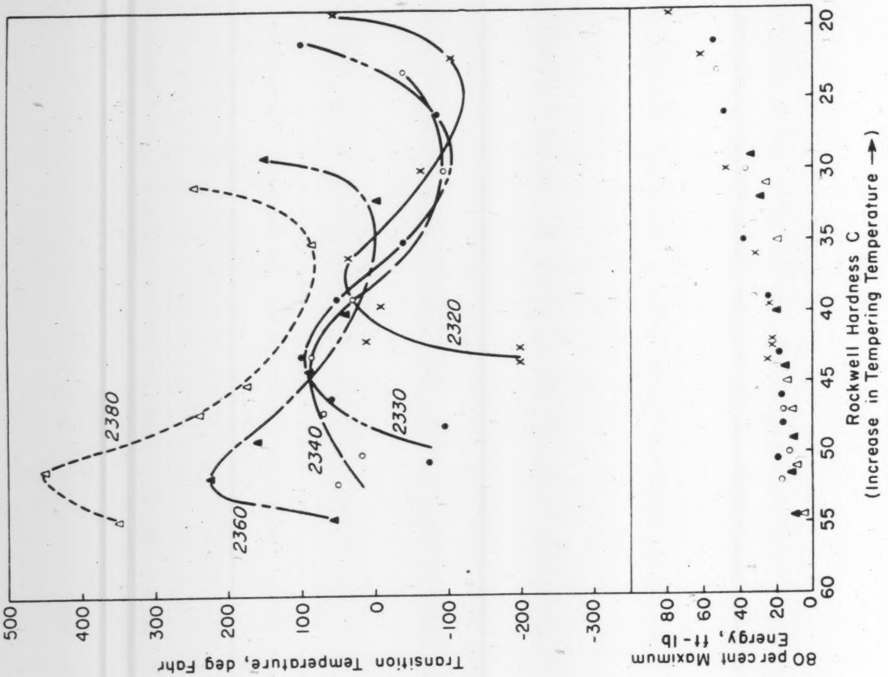

FIG. 10.—Transition Temperature and 80 per cent of Maximum Energy as a Function of Hardness and Carbon Content for the 2300 Series.

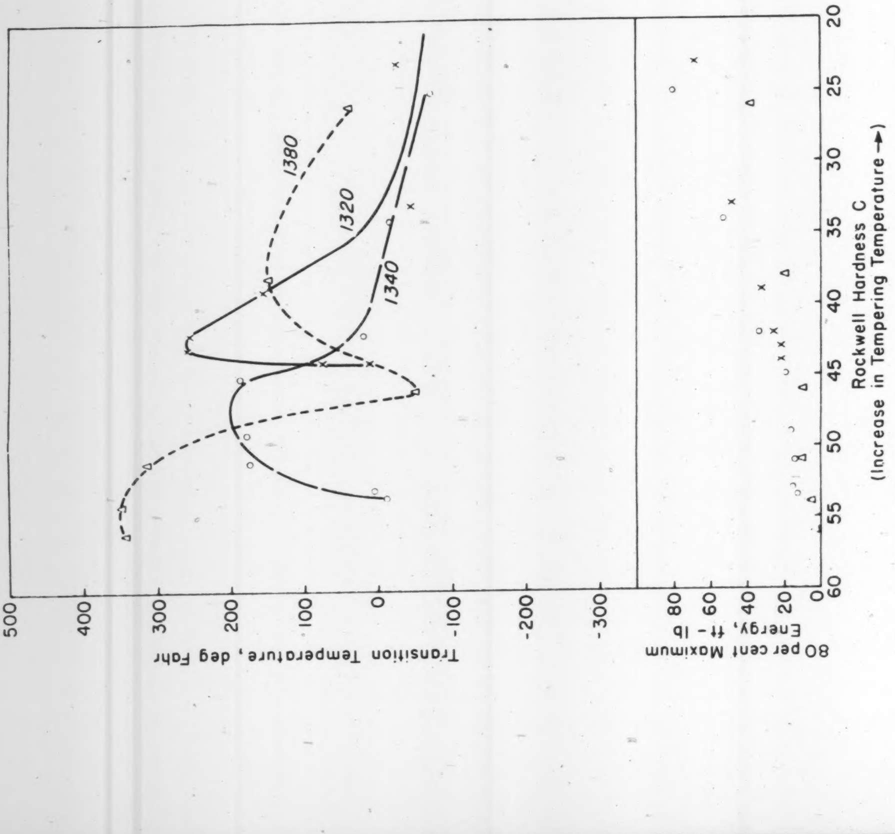

FIG. 9.—Transition Temperature and 80 per cent of Maximum Energy as a Function of Hardness and Carbon Content for the 1300 Series.

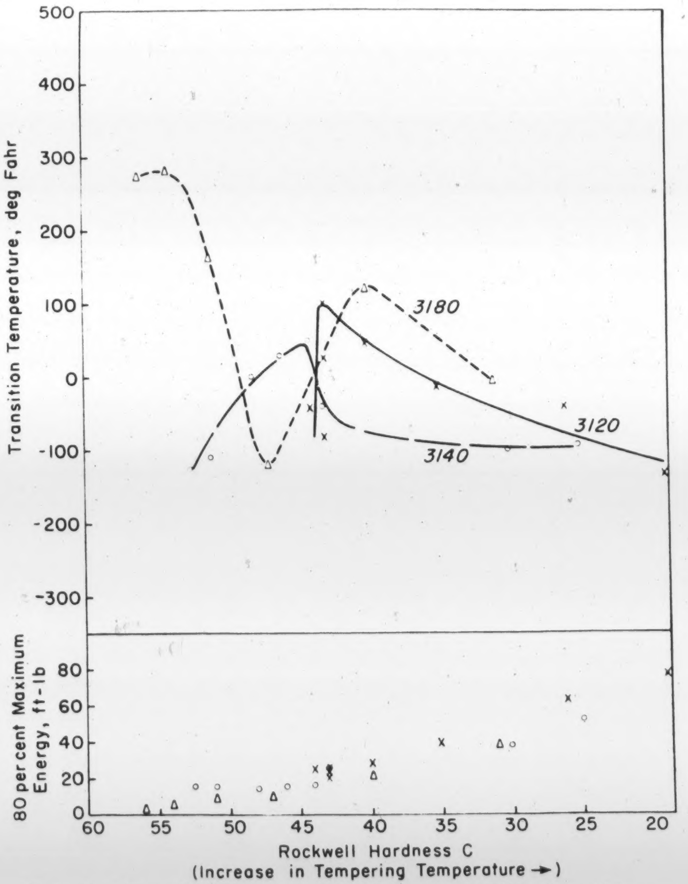

FIG. 11.—Transition Temperature and 80 per cent of Maximum Energy as a Function of Hardness and Carbon Content for the 3100 Series.

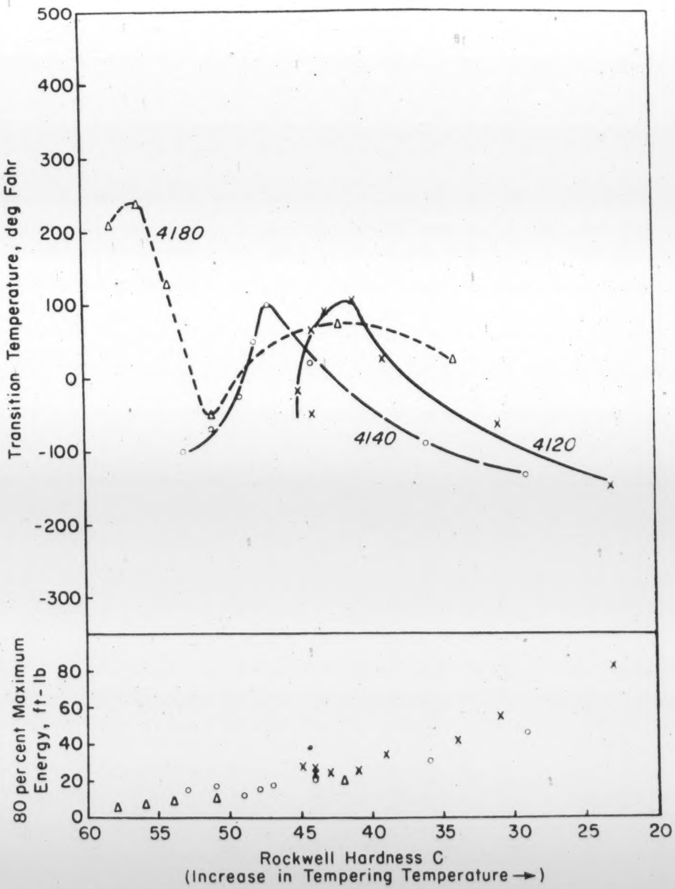

FIG. 12.—Transition Temperature and 80 per cent of Maximum Energy as a Function of Hardness and Carbon Content for the 4100 Series.

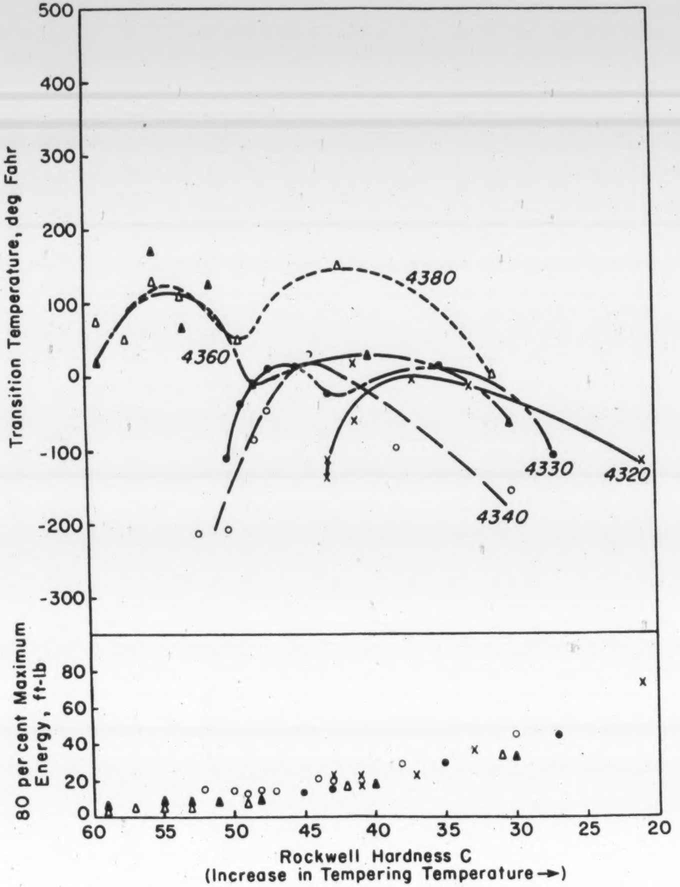

FIG. 13.—Transition Temperature and 80 per cent of Maximum Energy as a Function of Hardness and Carbon Content for the 4300 Series.

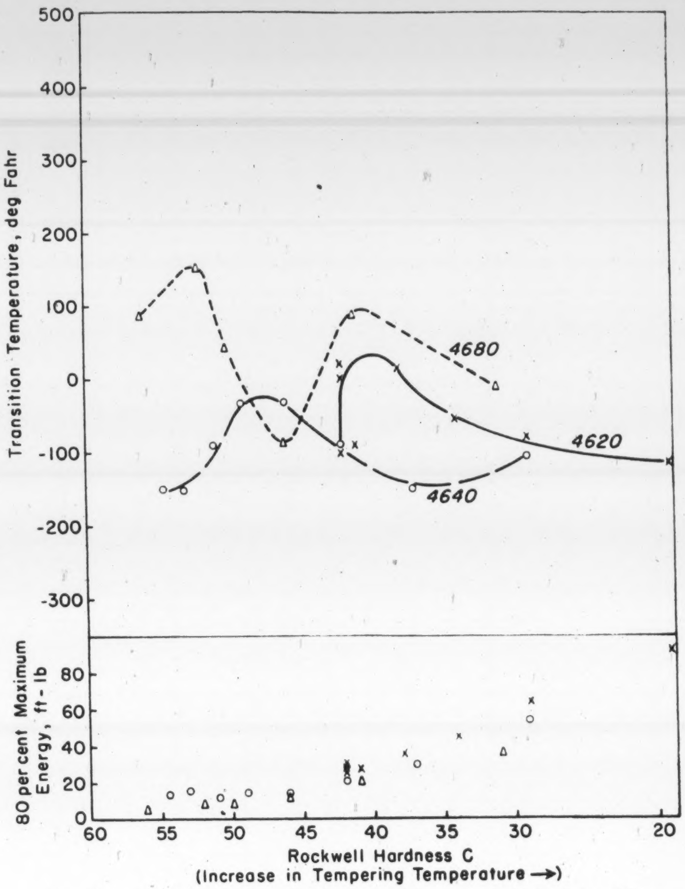

FIG. 14.—Transition Temperature and 80 per cent of Maximum Energy as a Function of Hardness and Carbon Content for the 4600 Series.

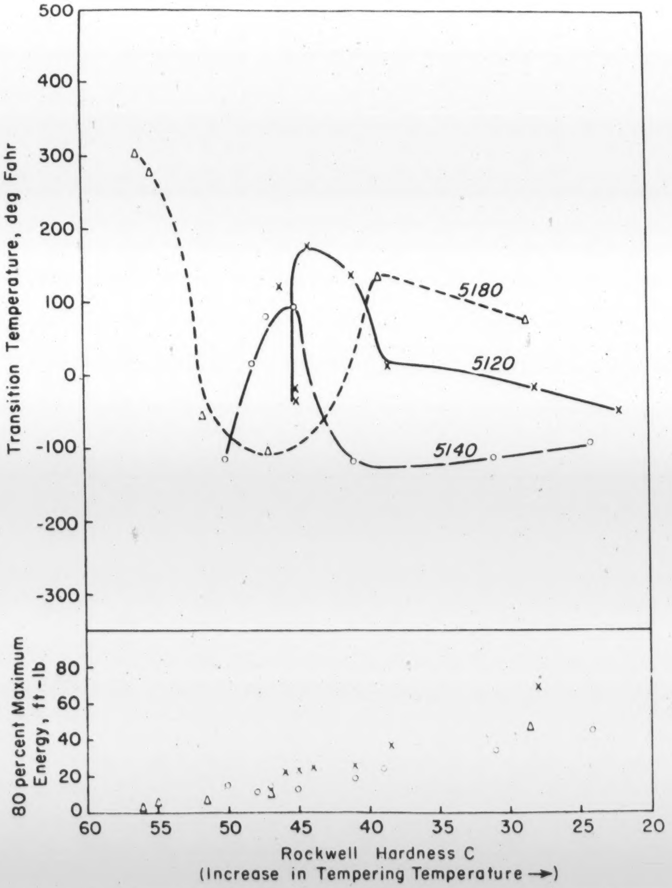

FIG. 15.—Transition Temperature and 80 per cent of Maximum Energy as a Function of Hardness and Carbon Content for the 5100 Series.

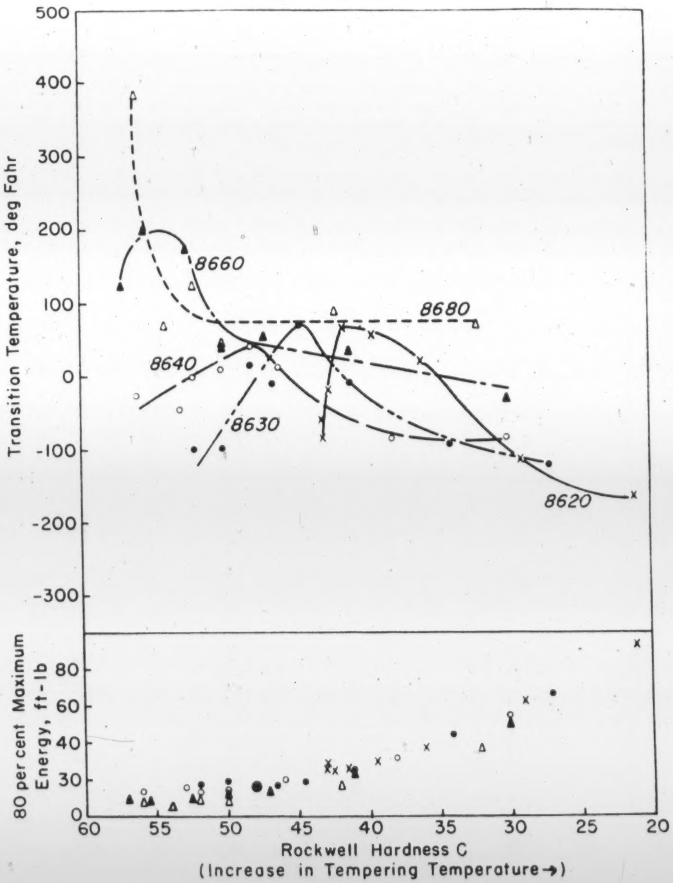

FIG. 16.—Transition Temperature and 80 per cent of Maximum Energy as a Function of Hardness and Carbon Content for the 8600 Series.

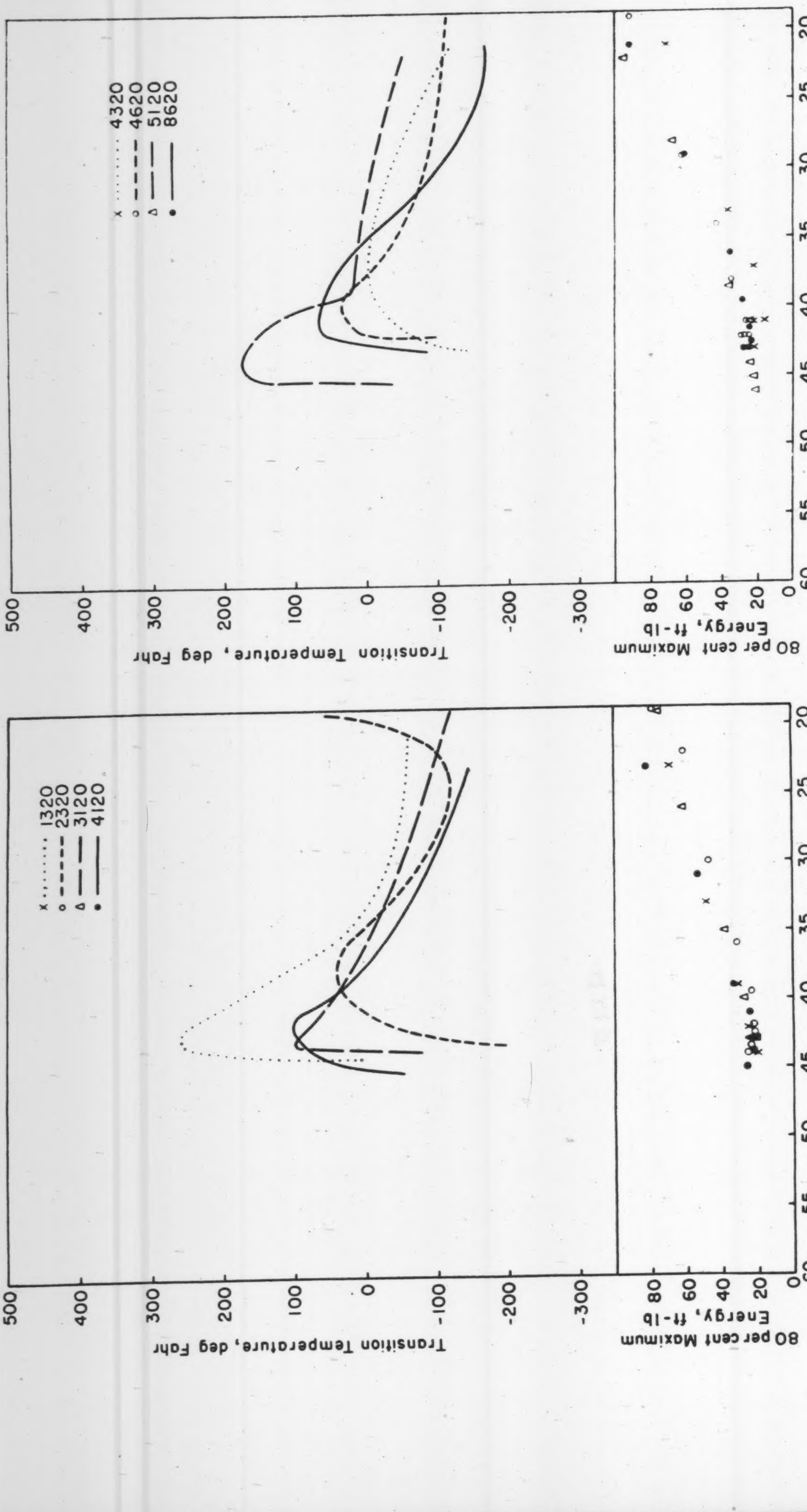

FIG. 17.—Variation of Transition Temperature and 80 per cent of Maximum Energy With Hardness for Various Grades of Steel at the 0.20 per cent Carbon Level.

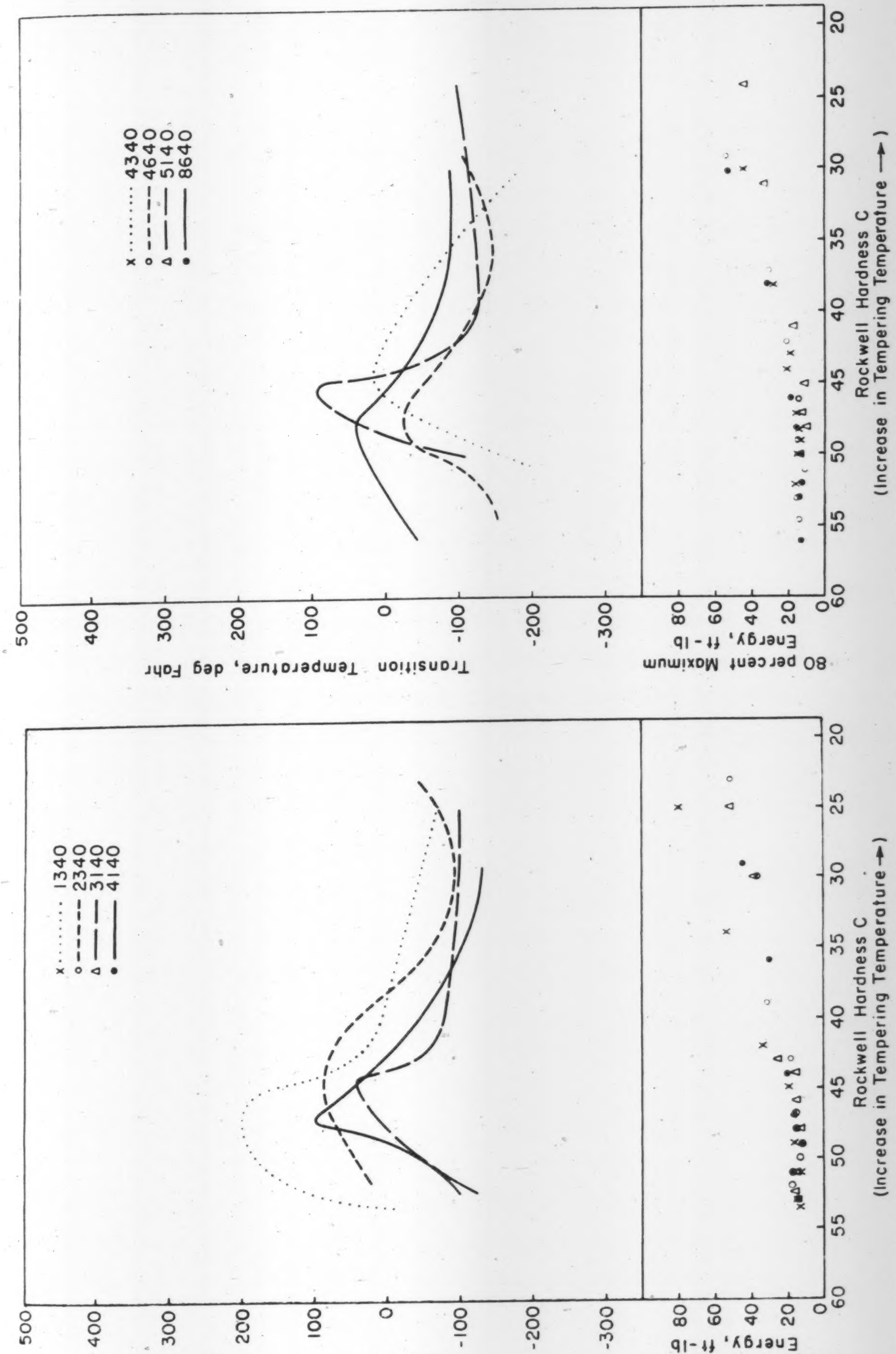

FIG. 18.—Variation of Transition Temperature and 80 per cent of Maximum Energy With Hardness for Various Grades of Steel at the 0.40 per cent Carbon Level.

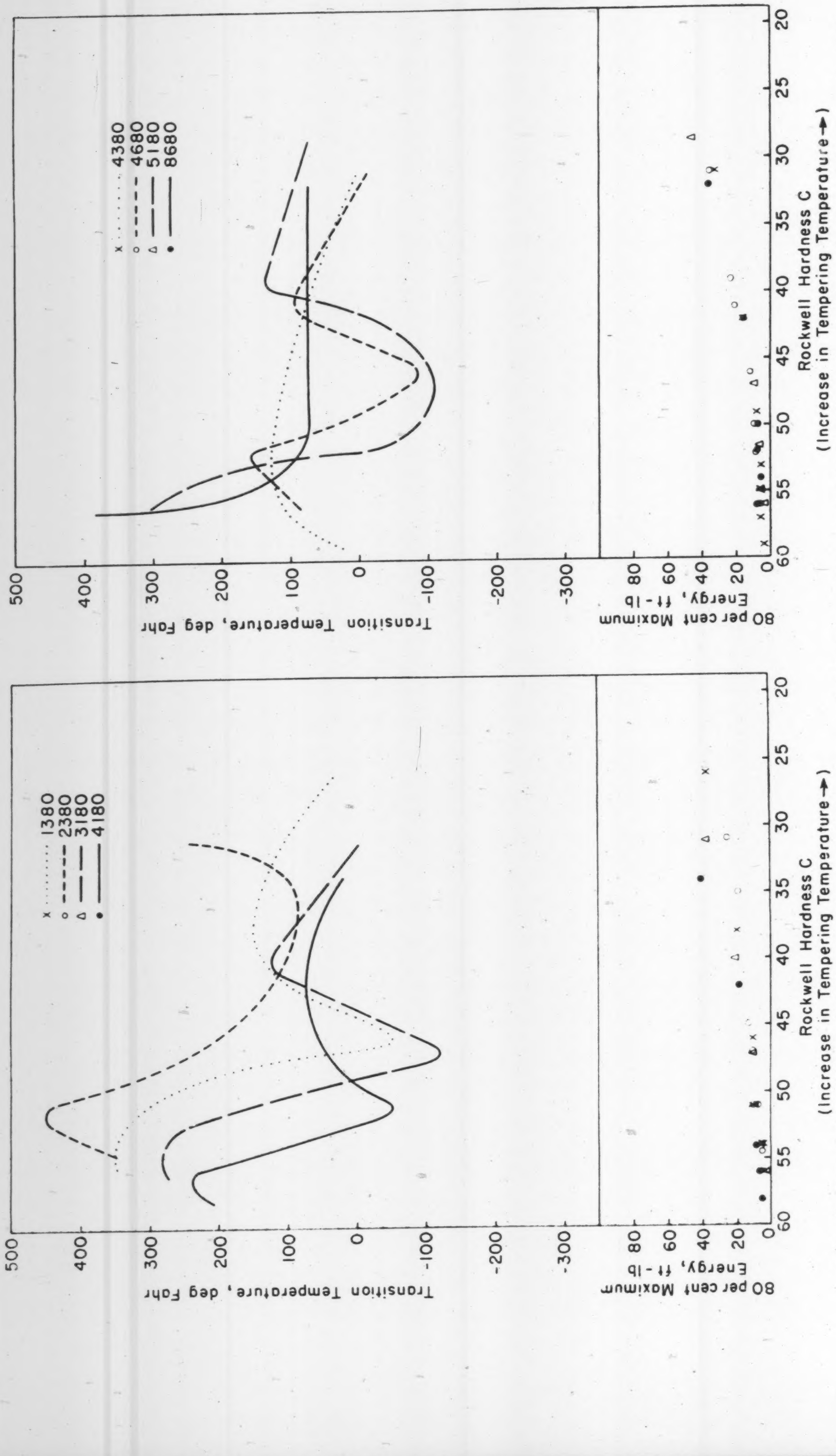

FIG. 19.—Variation of Transition Temperature and 80 per cent of Maximum Energy With Hardness for Various Grades of Steel at the 0.80 per cent Carbon Level.

The specimen blanks were austenitized for 30 min at the temperatures listed in Table II, oil quenched, tempered for 1 hr, then water quenched. Austenitizing and normalizing prior to machining were performed at the same temperature; this temperature was varied with carbon content in order to obtain fine grain sizes, which are also recorded in Table II. The hardenability of all the grades investigated was sufficient to harden the impact specimens completely.

The criterion of transition temperature used in this section is the temperature at 80 per cent of maximum energy.

Further experimental details can be obtained from the interim reports on this contract.

RESULTS AND DISCUSSION

Experimental results are presented in Figs. 1 to 19. Figures 1 to 8 present transition temperature as a function of tempering temperature; Figs. 9 to 16 present transition temperature and 80 per cent of maximum energy as functions of hardness with carbon content as the parameter, and in Figs. 17 to 19 with alloy grade as the parameter. (Figures 17 to 19 are presented in two parts, (*a*) and (*b*), to prevent crowding of the curves.)

The variation of transition temperature with tempering temperature may be discussed in relation to the various types of embrittlement exhibited by tempered martensite, namely, A-brittleness (so called 500 F embrittlement), B-brittleness, and temper brittleness. These various types of embrittlement are manifested by maxima in the curves of tempering temperature *versus* transition temperature.

A-Brittleness:

A-brittleness is that embrittlement exhibited by tempered martensites in the tempering range of 500 F to 800 F. Recent work (8) indicates that the cause of this embrittlement is the formation of a grain boundary cementite which manifests itself in the microscopic mechanism of fracturing which will be discussed in a later section. The strain-hardening exponent has been shown (9) to pass through a minimum in this tempering range.

Figures 1 to 19 reveal that all the steels exhibit A-brittleness. The amount of A-brittleness and the tempering temperature at which the A-brittleness peak occurs are functions of carbon and alloy contents. The amount of A-brittleness is greatest for the 0.80 per cent carbon heat in each grade.

The temperature at which the peak occurs varies at 0.40 per cent carbon level, for example, from 600 F to 800 F. For 4340 steel the peak is at the highest temperature, 800 F, and has the lowest transition temperatures at the low-tempering temperatures, that is, high-strength levels. The straight manganese 1300 grade exhibits the greatest amounts of A-brittleness in general, while the molybdenum-bearing grades exhibit the least embrittlement.

B-Brittleness:

It is believed that B-brittleness is caused by the formation of austenite upon tempering at 1200 F, which then transforms to martensite upon quenching from the temper or at a later time upon testing. The formation of austenite on tempering 2340 at 1200 F has been observed metallographically. Further support for this explanation of B-brittleness is provided by the work of Bleakney and Grosvenor (10), and Dube and Cunningham (11), who studied the phase changes of straight nickel steels.

B-brittleness is exhibited only by the 2300 series (Fig. 2), a straight nickel grade. Slight reversals are exhibited also by 4640 and 5140, but the 2300 series is the only one which showed this behavior as a grade and to such a large extent.

It should be borne in mind that this behavior in the 2300 series is not a property of tempered martensites in the same sense as for the other grades but is rather a manifestation of the presence of freshly formed martensite and that similar embrittlement would probably be obtained in the other grades at tempering temperatures high enough to actuate the same mechanism.

Temper Brittleness:

The particular type of brittleness known as temper brittleness is also observed in some of the curves of Figs. 1 to 19. Temper brittleness is developed in alloy steels by tempering in the range from 950 F to 1150 F for extended times or by cooling slowly through this range. The specimens used in the present study were tempered for 1 hr and quenched. Ordinarily, this treatment would develop very little, if any, temper brittleness. However, with the exception of the 2300 and 8600 series, the 0.80 per cent carbon heat of every grade of steel investigated exhibited a peak in transition temperature at a tempering temperature of 1000 F. Even in the 8600 series (Fig. 8), whereas the transition temperature for low-carbon contents decreased as the tempering temperature rose from 700 F, the transition temperature for 8660 did not decrease until the tempering temperature exceeded 1000 F, and for 8680 the transition temperature did not decrease at all. This peak at a tempering temperature of 1000 was generally not as high as the peak caused by A-brittleness at a tempering temperature of 500 to 700 F. Thus, it has been shown that a high-carbon content aggravates temper brittleness; grades of steel which developed no temper brittleness at low- and intermediate-carbon contents did develop temper brittleness in the 0.80 per cent carbon heats.

An examination of the temper embrittlement maxima obtained by tempering the 0.80 per cent carbon heats at 1000 F (Fig. 19 (*a*) and (*b*)) indicates the beneficial effect of molybdenum in minimizing temper brittleness. Grades 2380, 4380, and 8680 exhibit no maxima at all. Of these, 2380 is a straight nickel grade and the other two contain molybdenum. The remaining grades fall into the following order as regards amount of temper brittleness exhibited with 4100 showing the smallest peak and 5100 the largest: 4100, 4600, 1300, 3100, and 5100. The 4100 and 4600 grades contain molybdenum and the 1300, 3100, and 5100 do not. Thus, the three worst grades, insofar as temper brittleness is concerned, contain no molybdenum, while those grades which exhibit less or no temper brittleness do contain molybdenum; the straight nickel grades also exhibit no temper brittleness.

Transition Temperature versus Hardness and Alloy:

The curves of transition temperature *versus* hardness have the same general shape as those of transition temperature *versus* tempering temperature; however, they are displaced relative to one another because of the use of the different abscissa. Consider first the curves of Figs. 9 to 16. With the exception of the temper brittleness exhibited at a Rockwell hardness around C 40 in the 0.80 per cent carbon heats, the curves of transition temperature *versus* hardness are somewhat homologous within any one grade, the curves being displaced toward higher hardness as carbon content increases. This includes the A-brittleness peak which moves toward higher hardness as the carbon content increases. The height of this peak is the greatest for the 0.80 per cent carbon heat in each grade and is the lowest for the 0.40 per cent carbon heat in the 1300, 4600, 5100, and 8600 series. The height of the peak in the 0.20 per cent carbon heat is the lowest for the remaining grades with the exception of the 4100 series in which the 0.20 and the 0.40 per cent carbon heats have peaks of equal height.

The carbon content with the lowest transition temperature for any given hardness depends upon the particular hardness chosen. Although it is difficult to generalize, the best over-all compromise seems to be carbon contents of 0.40 and 0.30 per cent, which generally have the lowest transition temperatures at Rockwell hardnesses under about C 40 and above C 49. Exceptions are the low-carbon heats of the 2300, 4100, and 4300 grades which have lower transition temperatures as-quenched or tempered only slightly than steels of higher carbon contents at the same hardness.

Examine now Fig. 13, in which are presented the transition temperature data for the 4300 series. Although the transition temperature is a function of the hardness and the carbon content, the maximum energy (or 80 per cent of the maximum energy) is dependent only on hardness, within a narrow band. Within this band, at a given hardness, the energy generally increases as the carbon content decreases. It will be noted that the width of the band is much greater for most of the other grades, especially for the 1300 and 5100 series. It is generally true, however, that for any grade the impact energy increases as the carbon content decreases for any given hardness. The same sort of an analysis applied to Figs. 17 to 19 indicates that steels of various alloy grades, but of the same carbon content, likewise do not exhibit a unique relationship between 80 per cent of maximum energy and hardness, but that a band is obtained for this function. This is particularly true of the 0.20 and 0.40 per cent carbon heats. The 0.80 per cent carbon heats exhibit considerably less scatter in their 80 per cent of maximum energy *versus* hardness curve. The disposition within the band is different for these figures than for Figs. 9 to 16 in that the relative positions of the various alloy grades differ at the different carbon levels, whereas in Figs. 9 to 16 it was generally true that the lower the carbon the higher the energy for a given hardness.

The question of which alloy or combination of alloys best promotes low transition temperatures is one of vital interest to all engineers who design structures for low-temperature service. Stated in other words, the question is this: "For a given hardness and possibly for a given carbon content, which grade of steel will give the lowest transition temperature?" Examination of Figs. 17 to 19 indicates that, on the basis of transition temperature, there is no consistent rating that can be assigned to the various grades of steel at all carbon contents or hardness levels. Whereas at certain hardnesses there is very little spread in transition temperature for all grades of steel, that is, Rockwell C 34 in the 0.20 per cent carbon steels, at other hardness levels the spread may amount to several hundred degrees. Moreover, a grade that appears to be superior at one hardness level or carbon content may not be so at a different hardness or carbon content. For example, the 3½ per cent nickel grade 2320 has low transition temperatures over most of the hardness range, while this same grade at higher carbon contents is intermediate or inferior to other grades. Likewise, the nickel-chromium-molybdenum steel 8620 is superior at the lowest hardnesses but varies from intermediate to poor at other hardness levels and carbon contents. Similar relationships prevail for the various other grades tested. However, the four molybdenum-containing grades investigated, 4100 (chromium-molybdenum), 4300 (nickel-chromium-molybdenum), 4600 (nickel-molybdenum), and 8600 (nickel-chromium-molybdenum) series, among them had the lowest transition temperatures at most of the carbon contents and over most of the hardness range. Furthermore, at no carbon or hardness level does a molybdenum-containing steel have the highest transition temperature, or does a grade containing no molybdenum have the lowest transition temperature. As will be discussed later, the beneficial effect of molybdenum may be due to its role in offsetting the embrittling effects of phosphorus.

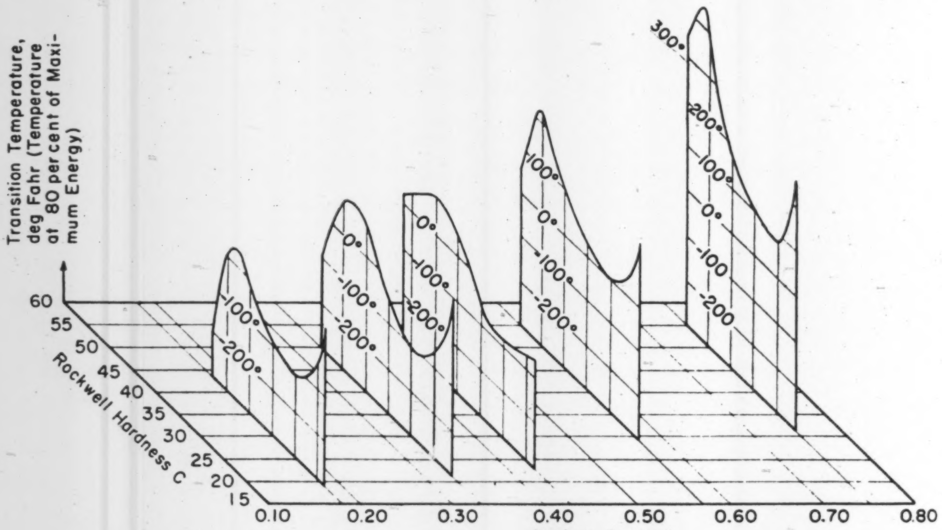

FIG. 20.—Interrelations Among Carbon Content, Hardness, and Transition Temperature for the 2300 Series.

The important conclusion to be reached from this discussion is that no one alloy grade is superior at all carbon and hardness levels, even within the limits of the present investigation. (It should be emphasized at this time that the curves apply to the particular heat treatment used in this investigation and that for a different heat treatment the curves would have a different relationship to one another. In particular, a tempering temperature treatment more conducive to temper brittleness might change the order of the curves at low hardnesses.)

One behavior exhibited by 0.20 per cent carbon alloy steels which can be observed in Fig. 17 is manifested by the nearly vertical portion of the curves at the as-quenched hardness. Tempering these steels at temperatures up to 600 F does not decrease the hardness but may, in fact, increase the hardness slightly; the transition temperature is, however, increased to a large extent by the tempering temperature.

Three variable plots summarizing the interrelations among hardness, carbon content, and transition temperature for the 2300, 4300, and 8600 series are presented in Figs. 20 to 22. These curves have already been adequately discussed previously. The three-variable representation is a convenient, clear method of presenting these data and for studying the relationships obtained.

COMMERCIAL HEATS

The discussion of the preceding section on the effects of alloy and carbon contents on transition temperatures applies

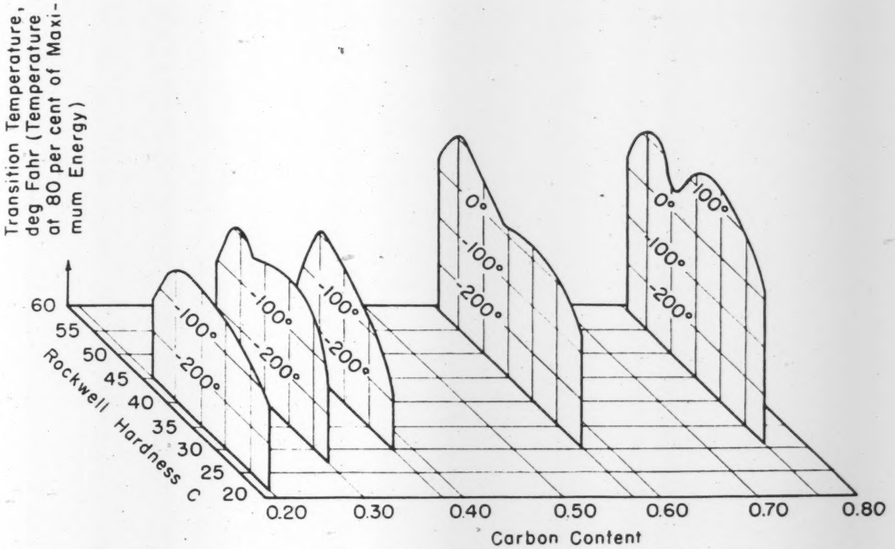

FIG. 21.—Interrelations Among Carbon Content, Hardness, and Transition Temperature for the 4300 Series.

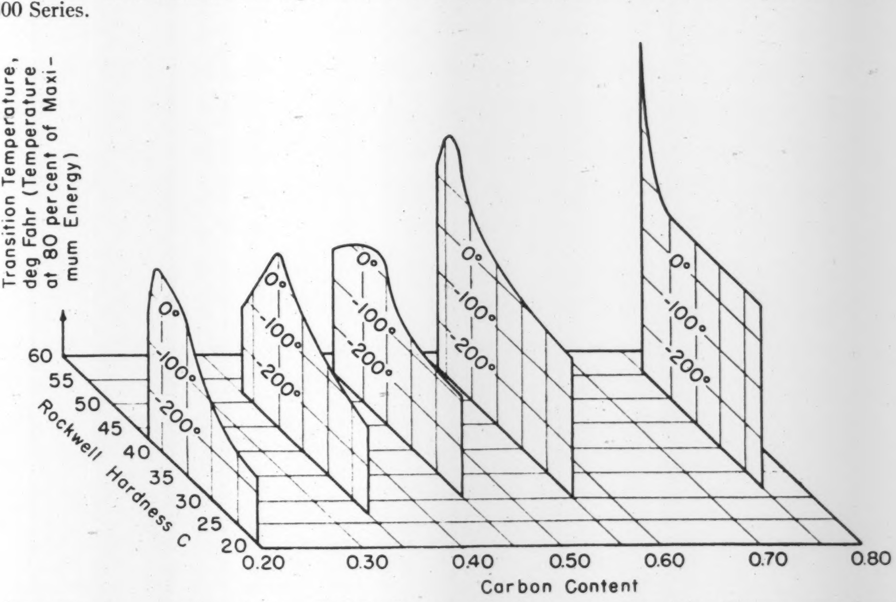

FIG. 22.—Interrelations Among Carbon Content, Hardness, and Transition Temperature for the 8600 Series.

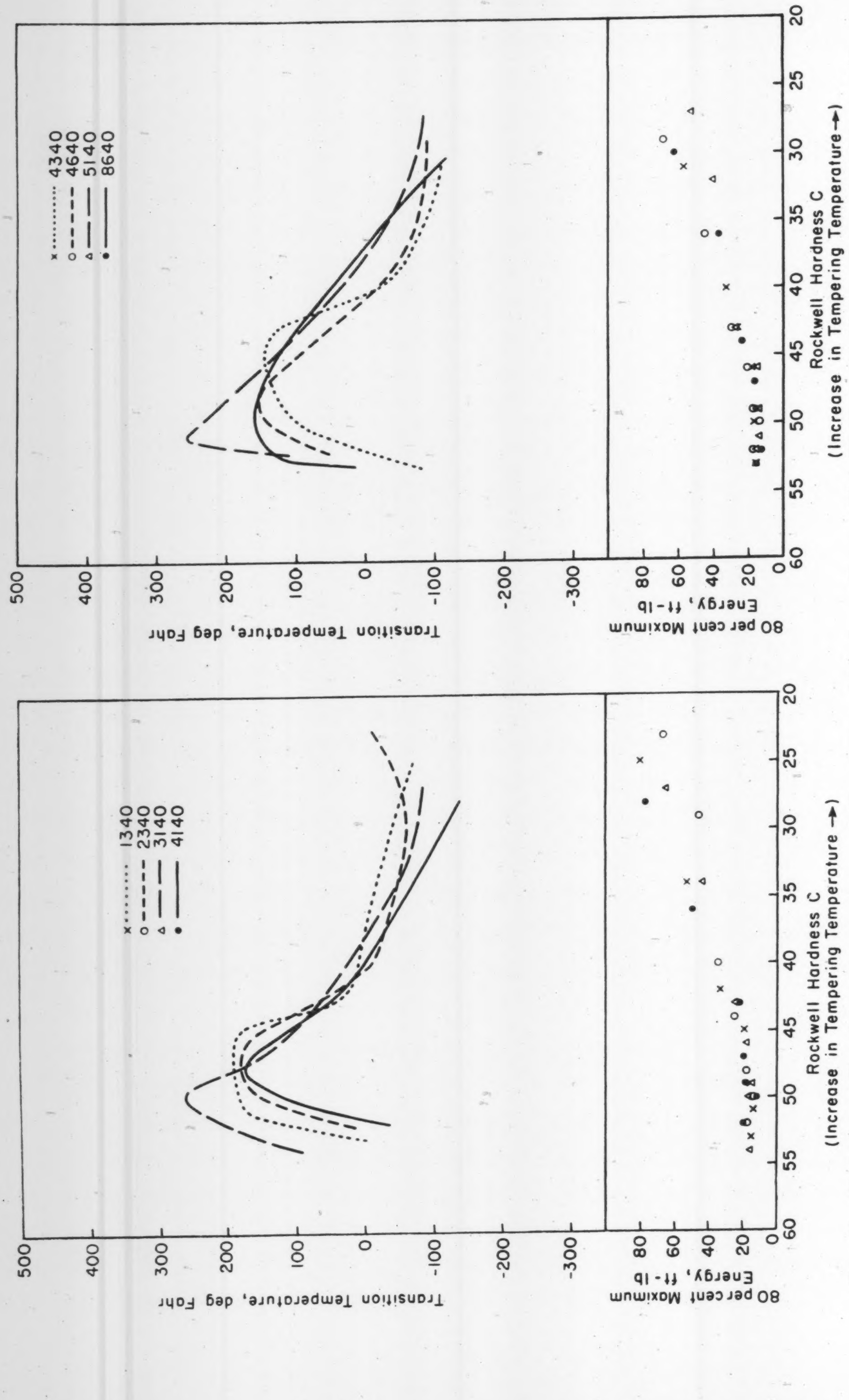

FIG. 23.—Variation of Transition Temperature and 80 per cent of Maximum Energy With Hardness for Various Commercial Heats of Steel at the 0.40 per cent Carbon Level.

to laboratory induction melted 500-lb heats with which it was attempted to maintain all conditions constant except the variables under investigation, namely, alloy and carbon content. As a part of this program, commercial heats of the eight grades of steel were examined in a like manner. Some of the heats were basic open-hearth and some basic electric-furnace heats. They were all of the same fine ASTM grain size,[4] in the heat-treated condition, as the laboratory heats, and contained 0.40 per cent carbon.

The transition temperature and values of 80 per cent of maximum energy are presented in Fig. 23 where they can be compared with the data on the laboratory heats presented in Fig. 18.

An examination and comparison of Figs. 23 and 18 reveals the following information:

1. 80 per cent of maximum energy transition temperatures are, as a group, higher for the commercial heats than for the laboratory heats.

2. Maximum energy values obtained above the transition range are also higher for the commercial steels than for the laboratory heats.

3. As in the case of the laboratory

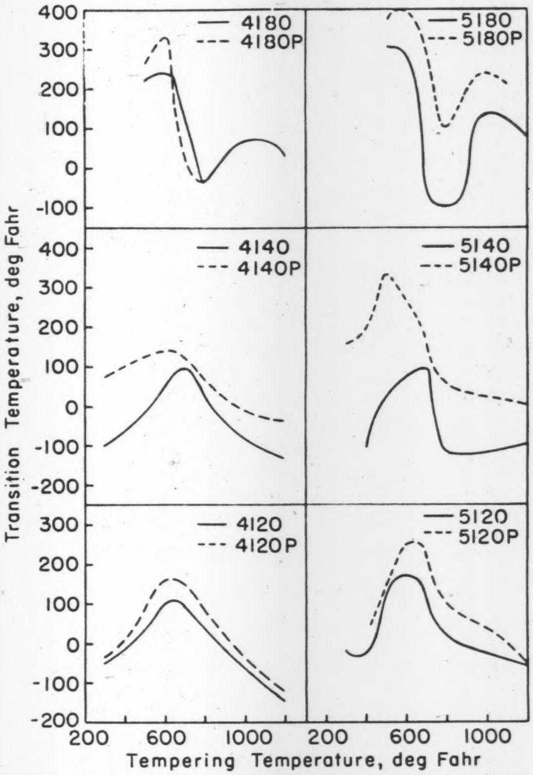

Fig. 24.—Effect of Phosphorus on Transition Temperatures for the 4100 Series Which Contains Molybdenum and the 5100 Series Which Has the Same Analysis as the 4100 Series Except That It Contains No Molybdenum.

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[4] Classification of Austenite Grain Size in Steels (E 19 – 46), 1955 Book of ASTM Standards, Part 1, p. 1602.

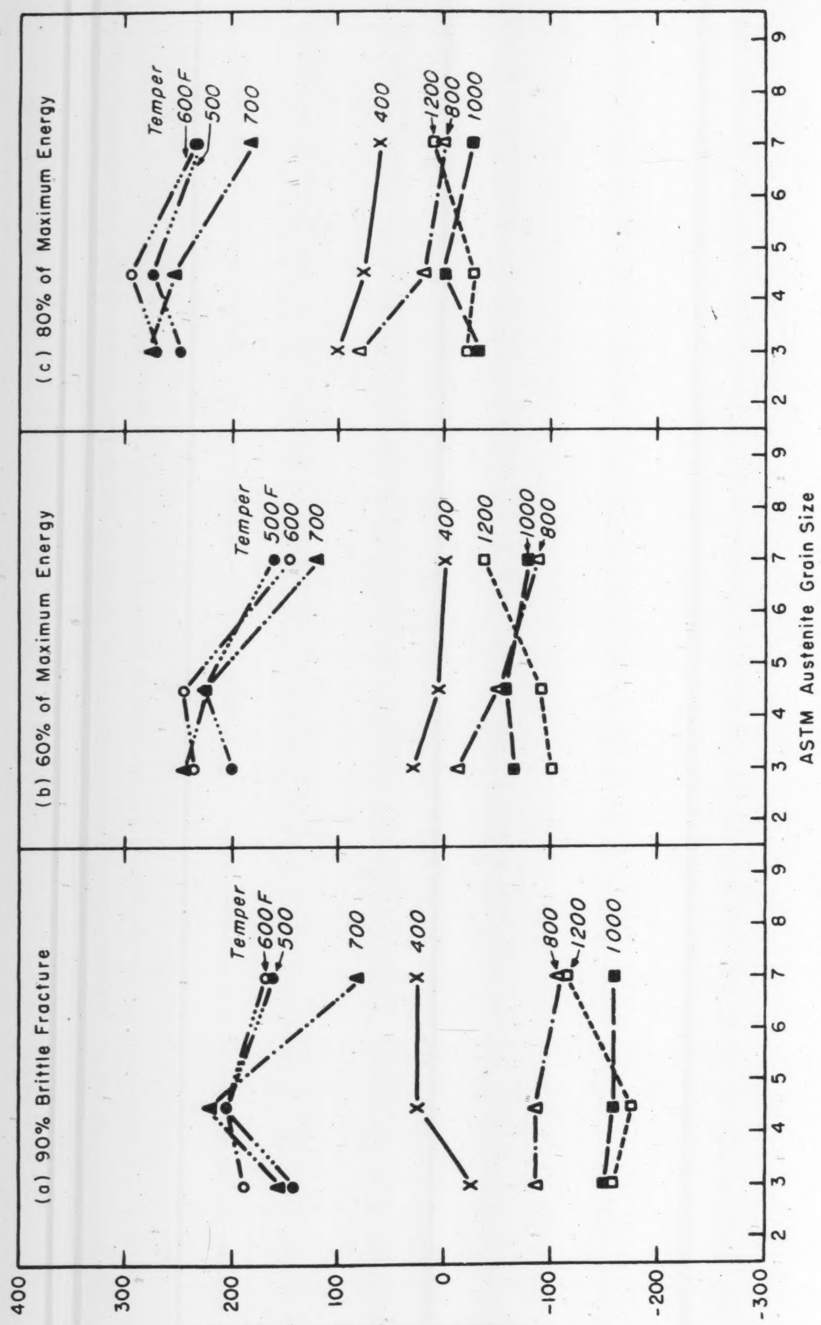


FIG. 25.—Effect of Austenite Grain Size on Transition Temperatures of 2340 Steel Q.

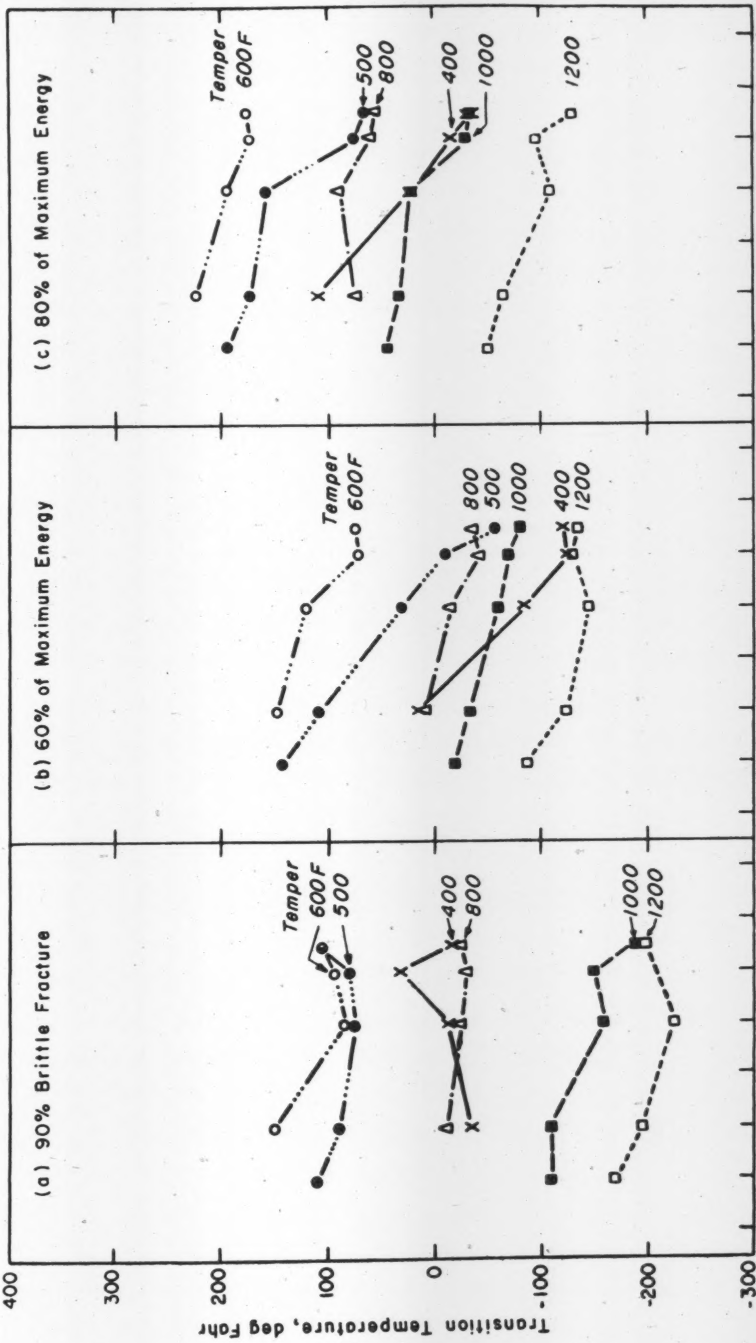


FIG. 26.—Effect of Austenite Grain Size on Transition Temperatures of 4140 Steel C.

steels, there is considerable variation in transition temperature among the various grades with different tempering treatments or hardnesses. However, in many respects, the data from the commercial heats bear out the data from the laboratory heats. Thus, 4340 is best at high and low hardnesses and the molybdenum-containing grades have the lowest transition temperatures over most of the hardness range. In other respects the agreement is not good, notably in the case of 5140 steel which rates poor in the commercial and good in the laboratory heats at the 0.40 per cent carbon level.

Phosphorus:

The effect of phosphorus content on the impact properties of two grades of steel at three carbon levels is presented in Fig. 24. The two grades investigated were 4100, containing 1 per cent chromium and 0.20 per cent molybdenum, and 5100, which is the same as 4100 except that it contains no molybdenum. The low phosphorus heats contained from 0.014 per cent to 0.023 per cent phosphorus, while the high phosphorus heats (signified by the letter "P") contained from 0.031 to 0.037 per cent phosphorus.

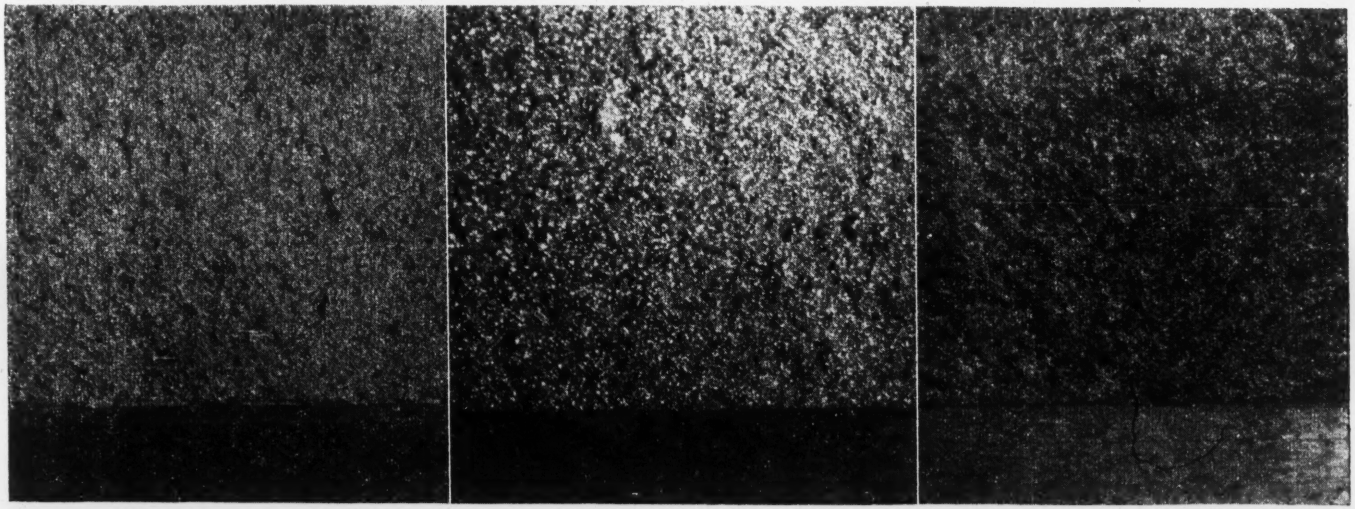
It can be seen that the added phosphorus has a deleterious effect on the transition temperature in every case except for grade 4180. It can further be seen that the transition temperature is elevated more for the steel containing no molybdenum, 5100, than for the steel containing molybdenum, 4100. Thus, molybdenum offsets the deleterious effect of phosphorus to a large extent. The low-phosphorus 4140 used in the present investigation has a molybdenum/phosphorus ratio of about 11, and the high-phosphorus 4140 a ratio of about 6.

EFFECT OF AUSTENITIC GRAIN SIZE

Previous investigations have yielded conflicting results as to the effect of austenite grain size on the transition temperature of quenched-and-tempered steels. As will be shown by the results of the present investigation, this discrepancy is attributable to the fact that austenite grain size may or may not affect the transition temperature of tempered martensite, depending upon the tempering treatment or hardness and upon the index selected for determination of the transition temperature.

The data obtained in the present investigation are presented in Figs. 25 and 26. Two grades of steel, 2340 and 4140, were investigated. The 2340 steel was a laboratory heat made by coarse grained practice (not aluminum killed) and the 4140 was a commercial heat made by normal fine grained practice. Austenitizing was for 30 min, tempering was for 1 hr, and the structure was tempered martensite throughout the specimen. All specimens were water-quenched after tempering.

The curves of transition temperature *versus* grain size have been drawn as point-to-point lines, so that the reader will have to imagine smooth trends where experimental scattering seems to indicate otherwise. In general, the data for grade 4140, Fig. 26, indicates decreasing transition temperature with decreasing grain size. The criterion of 90 per cent brittle fracture does not yield as clear a picture as the other two criteria in this case. The 60 and 80 per cent of maximum energy show the strongest dependence of transition temperature on grain size at tempering temperatures of 400 to 500 F, that is, at the highest strength levels investigated. Steel 2340, Fig. 25, appears to exhibit behavior similar to 4140. Whether the maxima exhibited by samples tempered at 500 F



(*a*) Coarse grain 2340 steel, tempered at 400 F. (*b*) Coarse grain 2340 steel, tempered at 500 F. (*c*) Coarse grain 2340 steel, tempered at 1200 F.

FIG. 27.—Macrographs of Brittle Fractures Showing the Sequence of Fracture for Tempering Below, Within, and Above the A-Brittleness Range (× 7.5). All broken at −320 F.

The intergranular fracture of (*b*) shows considerably more bright facets than the transgranular fractures of (*a*) and (*c*).

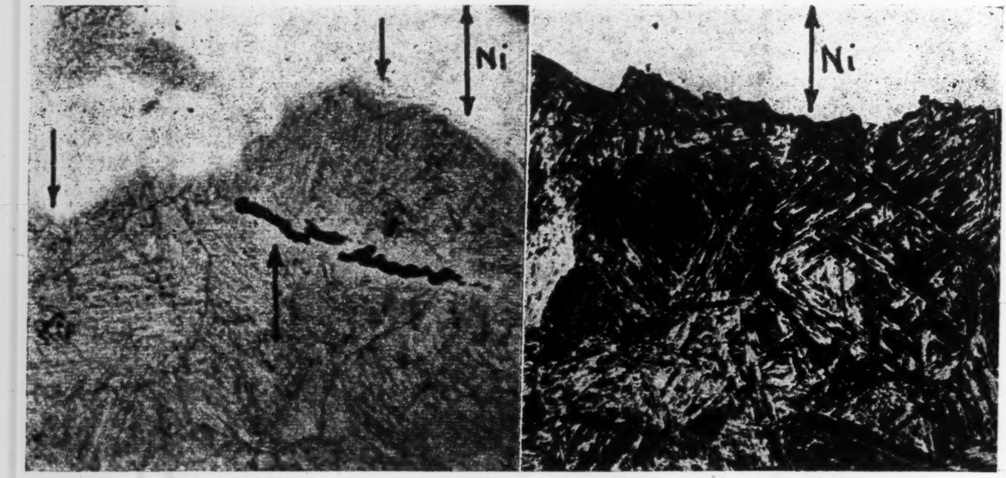


FIG. 28.—Laboratory 5140 Steel, Coarse Grain (X 250).

Austenitized ½ hr 1900 F, 5 min. 1550 F, oil-quenched. Tempered 1 hr at 400 F, water-quenched. Broken at −320 F.

Arrows indicated transgranular fracture with respect to prior austenite grain boundaries.

(*a*) Specimen fractured, then nickel-plated and cooled slowly from 1150 F to reveal prior austenite grain boundaries upon etching with zephiran chloride

(*b*) Nickel-plated specimen as broken. Etched in 2 per cent Nital.

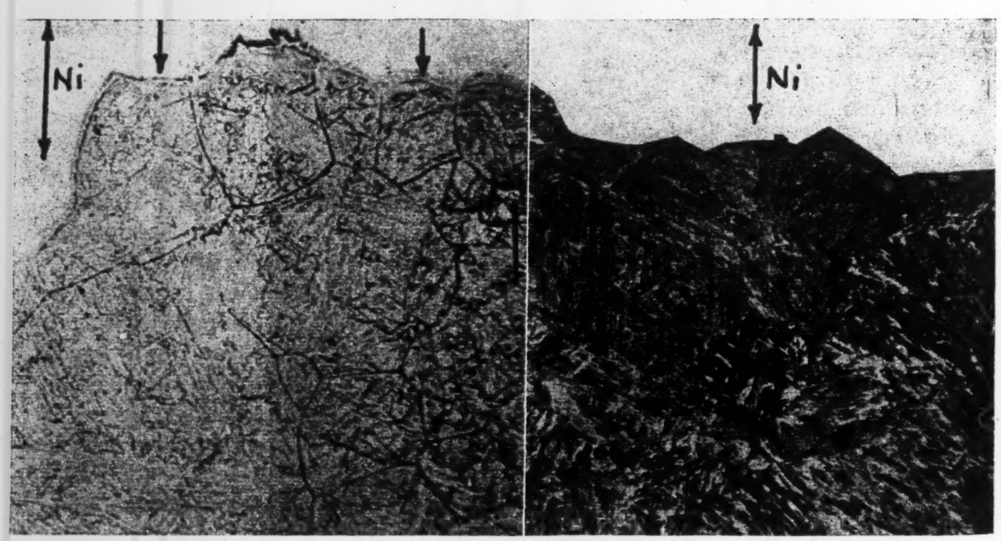


FIG. 29.—Same Steel and Treatment as in Fig. 28, Except Tempered 1 hr at 500 F (X 250).

Arrows indicate intergranular fracture along austenite grain boundaries.

(*a*) Nickel-plated and cooled slowly from 1150 F to reveal prior austenite grain boundaries. Etched in zephiran chloride.

(*b*) Nickel-plated specimen as broken. Etched in 2 per cent Nital.

or 600 F or 700 F is real or not is unknown. The 2340 does differ from the 4140 in respect to the samples tempered at 1000 F or 1200 F. The transition temperature of samples of 2340 tempered at 1000 F is not much affected by grain size, and those tempered at 1200 F exhibit a decreasing transition temperature with increasing grain size. The apparently unusual behavior is associated with the B-brittleness exhibited by steel 2340. The coarsened austenite did not exhibit B-brittleness in specimens tempered at 1200 F while the finer grained austenite did. This accounts for the behavior which is contrary to that usually observed.

MICROSCOPIC FEATURES OF FRACTURING

It is well known that brittleness revealed by the impact test may have entirely different structural origins in the steel and yet look the same, not only in the absorbed energy but also to a degree in the macroscopic appearance of the fractures. Tempered martensite has a very uniform microstructure, but the appearance of impact fracture in this structural state varies widely with hardness. Also, unusual degrees of brittleness may be induced by some customary tempering cycles, as well as by others not so common in commercial practice. With the exception of temper-embrittled martensite, little is known of the relation between the microstructures of the tempered martensite and the features of the fracture that are responsible for the macroscopic brittle appearance. In view of this, impact fractures of tempered martensite were examined

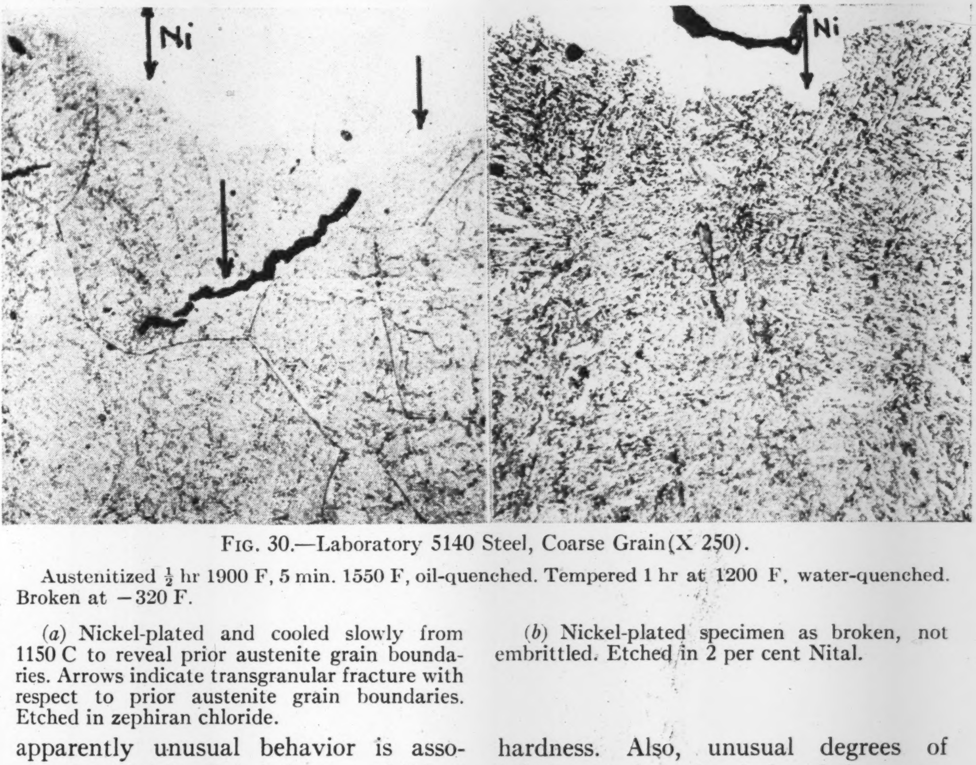


FIG. 30.—Laboratory 5140 Steel, Coarse Grain (X 250).

Austenitized $\frac{1}{2}$ hr 1900 F, 5 min. 1550 F, oil-quenched. Tempered 1 hr at 1200 F, water-quenched. Broken at −320 F.

(*a*) Nickel-plated and cooled slowly from 1150 C to reveal prior austenite grain boundaries. Arrows indicate transgranular fracture with respect to prior austenite grain boundaries. Etched in zephiran chloride.

(*b*) Nickel-plated specimen as broken, not embrittled. Etched in 2 per cent Nital.

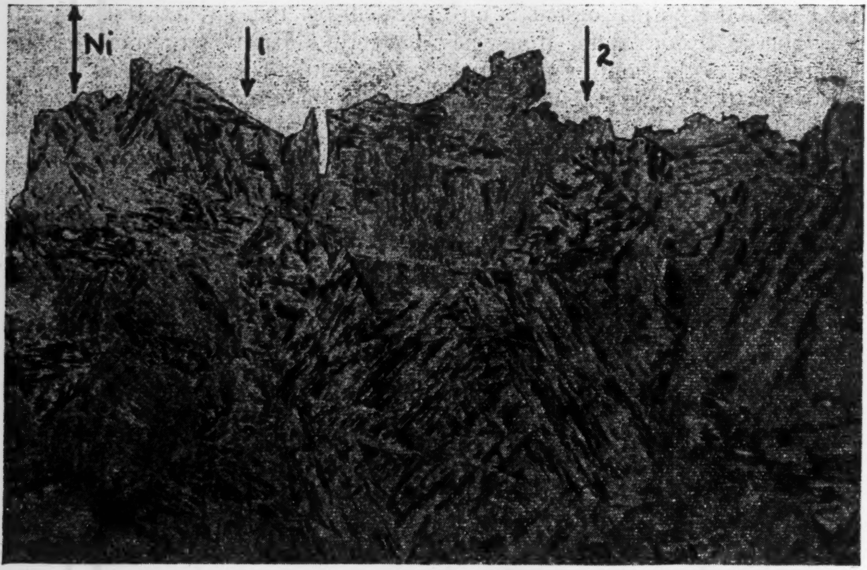


FIG. 31.—Final Path of Fracture of Coarse Grain 2340 Steel. Etched in 2 per cent Nital (× 250).

Tempered 1 hr at 400 F, and broken at −320 F. The fracture path consists of transgranular cleavages and ductile partings, arrows Nos. 1 and 2, respectively.

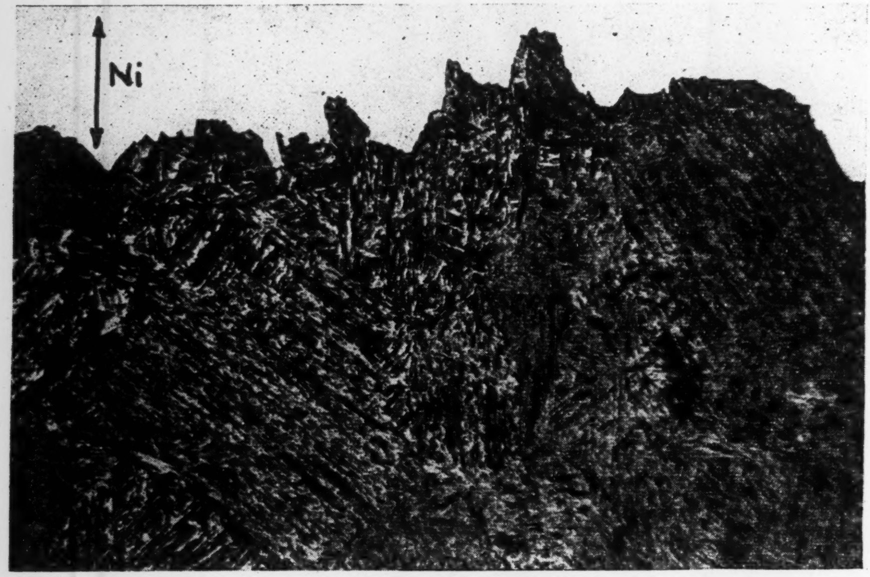


FIG. 32.—Transgranular Fracture Path of Coarse Grain 2340 Steel. Etched in 2 per cent Nital (× 250).

Tempered 1 hr at 400 F and broken at 212 F. Although the fracture surface shows nearly 100 per cent cold work, the depth of deformed metal appears to be very shallow.

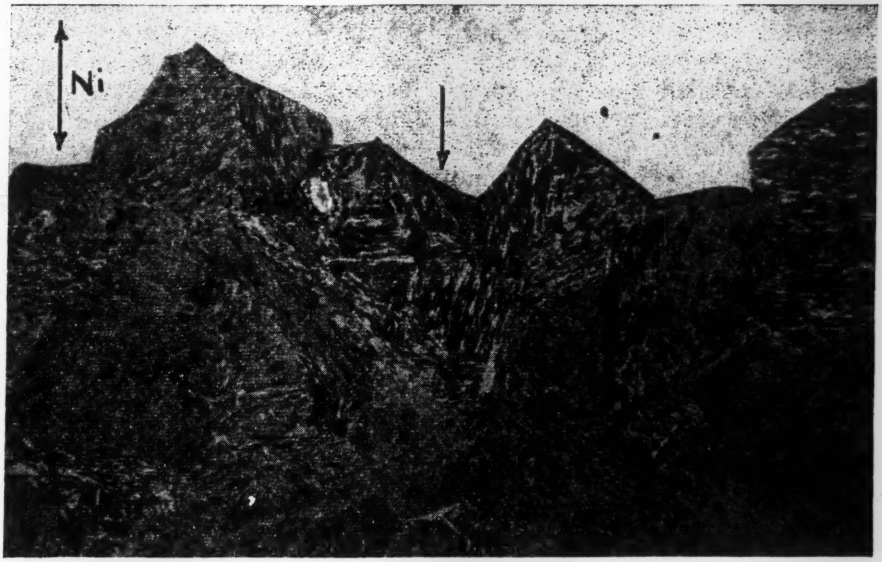


FIG. 33.—Intergranular Fracture Path Along Prior Austenite Grain Boundaries of Coarse Grain 2340 Steel. Etched in 2 per cent Nital (× 250).

Tempered 1 hr at 500 F and broken at −320 F.

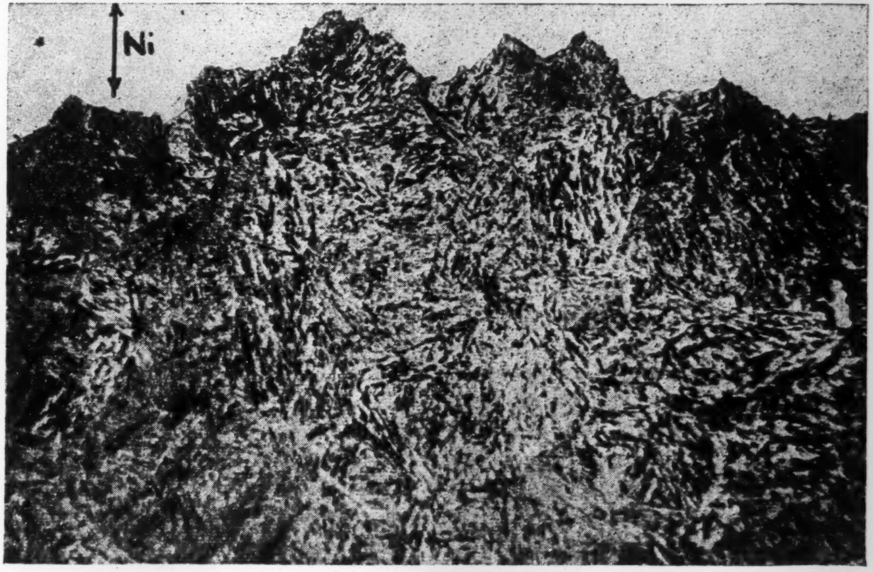


FIG. 34.—Transgranular Fracture Path of Coarse Grain 2340 Steel. Etched in 2 per cent Nital (× 250).

Tempered 1 hr at 500 F, and broken at 500 F.

with the object of determining microscopically the features that constituted their macroscopically brittle character.

The as-austenitized grain size had no effect on the essential features of the fractures other than to enlarge structural components such as prior austenite grains and martensite spines. In view of this, illustrations presented in Figs. 27 to 36 were made from fractured specimens where the as-austenitized grain size was ASTM Nos. 2 to 4.[4]

In the soft martensites tempered at 1000 F and above, the transitions from 100 per cent brittle to 100 per cent ductile fracture roughly span the energy of transition. The fracture transition takes place by restriction of the brittle region to a smaller and smaller area until the brittle region disappears entirely at the higher testing temperature end of the transition. On the other hand, in relatively hard martensite, a macroscopically observable fracture transition does not occur, at least not in the sense that the term has been used in designating the fracture transition in soft steel. Instead, the bright facets that indicate brittleness diminish in number per unit area as the testing temperature is increased. Between these two extremes of soft- and hard-tempered martensites, there is a gradual change in the type of fracture in keeping with absorbed energy changes.

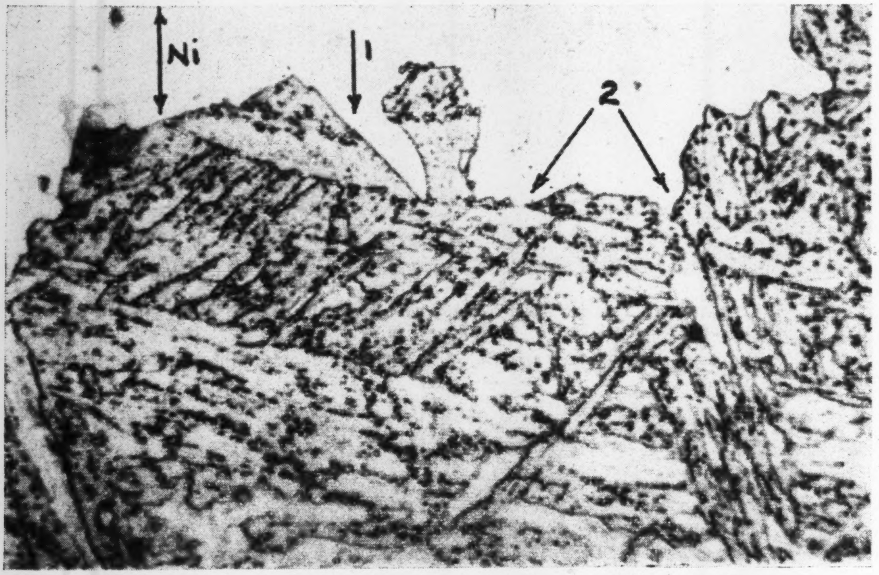


FIG. 35.—Coarse Grain 2340 Steel. Etched in 2 per cent Nital (× 2000).

Tempered 1 hr at 1200 F, and broken at −320 F.

Arrow No. 1 shows ferrite cleavages, and arrows No. 2 indicate ferrite grain boundaries that participated in the fracture path.

One of the more striking observations of the fractured surfaces is the change in appearance of the facets in brittle specimens according to whether they are tempered below, within, or above the A-brittleness range. Figure 27 shows the low-temperature fractured surfaces of specimens tempered at 400, 500, and 1200 F, respectively. The sequence of fracture from transgranular, to intergranular to transgranular, with respect to prior austenite grain boundaries, will be discussed later.

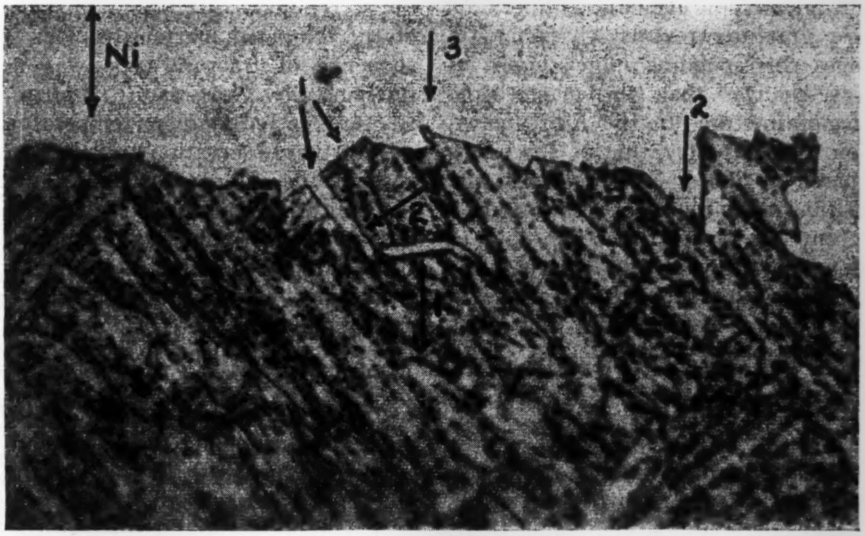


Fig. 36.—Same Sample as Fig. 35 Showing a Different Area. Etched in 2 per cent Nital (× 2000).

Arrow No. 1 indicates ferrite cleavage, No. 2 indicates fracture along ferrite grain boundaries, and No. 3 indicates transgranular cleavage across a number of similarly oriented ferrite grains.

Specimens were sectioned normal to the fracture surface and examined along the path of fracture. In order to make these observations, the specimens were plated with nickel. This procedure protected the fracture during preparation of the metallographic section. Additional studies were made to compare microscopic rating of per cent brittleness with macroscopic estimates of brittle fracture.

The brittle fracture ratings for the various tempered martensites and testing temperatures are shown in Table III.

The microscopic data were obtained by observations through a No. 5 grid Bausch & Lomb grain-size eyepiece, and then the per cent brittleness was estimated for each field across the fracture from the notch to the bottom of the specimen. These observations were made at magnifications of 150 to 300 ×, depending upon the structures involved. Brittleness was arbitrarily defined in these measurements as cleavages or straight fracture partings, while torn or irregular fracture paths were considered ductile.

TABLE III.—MICROSCOPIC EVALUATION OF FRACTURES.

Specimen[a]	Tempering Temperature, deg Fahr	Testing Temperature, deg Fahr	Energy, ft-lb	Visual Brittle, per cent	Micro Brittle, per cent
QB1-19......	400	−320	4½	100	51
QB1-12......	400	+212	16	80	11
QB1-13......	400	+400	17	...	4
QB2-19......	500	−320	1½	100	87
QB2-11......	500	+85	6	90	49
QB2-4.......	500	+300	15	...	18
QB3-2.......	600	−320	1½	100	82
QB3-10......	600	+150	8	90	58
QB3-3.......	600	+600	18	...	13
QB5-14......	800	−230	3	100	58
QB5-13......	800	+50	24	50	32
QB5-10......	800	+300	29	0	5
QB6-13......	1000	−230	4	100	67
QB6-5.......	1000	−50	29	50	27
QB6-2.......	1000	+200	42	0	11
QB7-11......	1200	−320	5	100	74
QB7-13......	1200	−120	38½	70	53
QB7-10......	1200	+200	62½	0	13

[a] Laboratory 2340 coarse grain.

Prior heat treatment consisted of austenitizing ½ hour at 2000 F, equalizing 5 min at 1550 F, and oil quenching. Tempering time was 1 hr.

The microscopic evaluation of per cent brittle fracture reveals characteristic changes corresponding more closely to energy changes than the macroscopic rating of fracture. In the A-brittleness region, microscopic rating of specimens tempered at 500 F and broken in the transition range revealed 87, 49, and 18 per cent brittle fracture from low to high testing temperature. This is to be compared with micro-brittle ratings of 51, 11, and 4 per cent for specimens tempered at 400 F.

Effect of Tempering Temperature Upon Fracture Path:

In 100 per cent brittle fractures, the relation of fracture path to steel structure depends upon the tempering treatment that has been given to the martensite. The characteristic changes of fracture path, with respect to the prior austenite grain boundaries, are illustrated by Figs. 28 to 30. These specimens were broken in the usual manner after the indicated treatment. The specimens were then sectioned and nickel plated. One-half was examined after etching with nital, and the other was temper embrittled in order to reveal more clearly the as-austenitized grain boundaries. Thus, upon etching with zephiran chloride reagent, the prior austenite grain boundaries were clearly delineated, and the paths of fracture, with respect to this feature of microstructure, became evident. Of course, as the temper embrittlement took place after the specimen was broken, it had nothing to do with the nature of the fracture.

The fractures, illustrated by Figs. 28 to 30 show a progression from transgranular fracture after tempering at 400 F, to intergranular after tempering at 500 F, and again to transgranular after tempering at 1200 F. Transgranular and intergranular refer to the austenite grains present just prior to quenching, not to the derived ferrite grain structure of the tempered martensite. This same sequence of fracture type was noted previously by Grossmann (12) in impact fractures tested at room temperature.

A further observation was made in the case of the intergranular path of 100 per cent brittle fractures in the A-brittleness range. Not only was the main fracture path predominantly along grain boundaries, but subsidiary cracks at the grain boundaries were common.

Influence of Testing Temperature Upon the Fracture at Given Hardness Levels:

Up to this point, the discussion has been concerned with 100 per cent brittle fractures. Increasing the testing temperature brought about more evidence of ductility in the "brittle" portion of the fractures, and, in some instances, a change in the microstructural features that participated in the fracture path. As might be anticipated from the dependence of the 100 per cent brittle fracture type upon the tempering temperature, the evidence of increasing ductility, with rise in testing temperature, likewise depends upon the hardness of the tempered martensite. The illustrations to follow will show the influence of increasing the testing temperature at a series of hardness levels.

Below the A-Brittleness Range.—As previously noted, macroscopically 100 per cent brittle fractures of martensite tempered for 1 hr at 400 F display paths of rupture that are predominantly transgranular with respect to prior austenite grains. Numerous flat transgranular facets give the fracture its brittle appearance, although some evidence of ductility is present even in specimens broken at −320 F. This is illustrated in Fig. 31. As recorded in Table III, 51 per cent of the fractured surface of the specimen tempered at 400 F and tested at −320 F is brittle by microscopic measurements. As the testing temperature is raised, deformation at the fractured surface increases gradually, until at 212 F only 11 per cent of the fracture is brittle, as illustrated in Fig. 32. Concomitantly, there is a decrease in the number of plane transgranular facets per unit area of macroscopically brittle area.

The microscopic observations confirm the macroscopic appearance. The plane transgranular facets give the fracture a brittle appearance over a considerable range of testing temperature. As the plane transgranular facets disappear, the appearance of the central regions of the fractures of the specimens broken at successively higher temperatures slowly become less brittle without ever exhibiting an abrupt fracture transition. As the fracture transition is gradual, so also is the rise in energy with increase in the testing temperature. The disappearance of the last of the plane transgranular facets from the fracture corresponds in general to the levelling out of the energy curve at the maximum value. In the particular set of specimens used in the illustrations in Figs. 31 and 32, this occurs between 150 F and 200 F on the testing temperature scale. At about the same testing temperature, the fracture becomes macroscopically unreadable. On the last appearance of plane facets, the central area on which they are distributed constitutes about 75 per cent of the surface of the fracture.

Within the A-Brittleness Range.—As previously noted, the 100 per cent brittle fractures of martensite tempered within the A-brittleness range were predominantly intergranular with respect to the prior austenite grains. With increase in testing temperature, the intergranular type of fracture persists, giving way but slowly to fractures that show evidence of cold work. The two extremes of the testing temperature series are illustrated by Figs. 33 and 34. In Fig. 33, at a testing temperature of −320 F, the fracture is almost entirely intergranular. In Fig. 34, at a testing temperature some 800 F higher, the fracture shows the same sort of superficial deformation in the immediate vicinity of the rupture that is noted in the specimens tempered at 400 F. The energy curve for the particular series is just approaching its maximum at the testing temperature of 500 F.

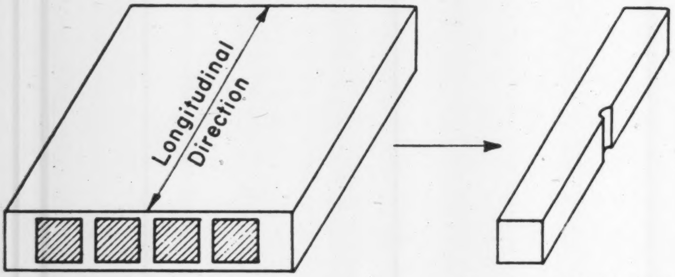
Thus, A-brittleness is characterized by intergranular fracture at all testing temperatures where bright facets appear on the fractured surface, and, in this respect, differs from the fractures of specimens tempered at 400 F. On the other hand, the evidences of ductility that come about gradually with increase in testing temperature are microscopically indistinguishable from those observed with specimens tempered at 400 F. On attainment of the maximum energy in the energy-testing temperature relation the ruptures of specimens tempered at 400 F and 500 F are alike in microscopic appearance, showing but a very superficial cold-worked region immediately adjacent to the path of rupture. The path of the ductile rupture is transgranular. Thus, in the specimens tempered at 400 F, both brittle and ductile rupture are transgranular, while on tempering at 500 F, there is a change in the microstructural features that participate in the rupture, for the brittle rupture is intergranular and the ductile rupture is transgranular.

Above the A-Brittleness Range.—At tempering temperatures above A-brittleness, the 100 per cent brittle fractures are characterized by a mixture of intergranular and transgranular brittle partings.

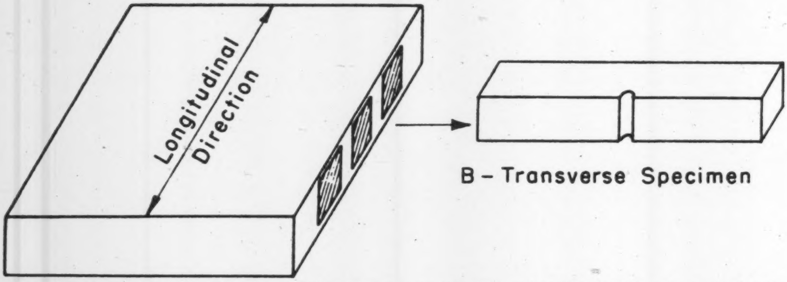
In martensite tempered at 1000 F and 1200 F, the brittle fractures continue to be transgranular with respect to the prior austenite grains, as shown in Figs.

35 and 36. There is, however, a marked shortening of the interval of testing temperature required to bring about the transition from 100 per cent brittle to a completely ductile fracture, and this is accompanied by macroscopically evident restriction of the region of the fracture in which plane transgranular facets occur. These changes in fracture appearance take place gradually as the tempering temperature is raised from 800 F to 1200 F. The impact fractures change gradually from those wherein the central brittle appearance die away rather slowly with increase in testing temperature to those wherein the remaining brittle portion of the fracture remain brittle in appearance but are restricted to a smaller central area of the fracture until the brittle area disappears entirely.

Increase of tempering temperature from 800 F to 1200 F causes a gradual rise in the maximum energy level, a shift in the transition toward lower testing temperatures, and more abrupt energy and fracture transitions. The increase in maximum energy is explained by the increase in the depth of the cold-worked metal that accompanies fracture above the energy transition and by the increasing area of the fracture forming the peripheral region of ductile fracture within the energy transition range.



A-Longitudinal Specimen



B-Transverse Specimen

FIG. 37.—Geometry of Specimens With Respect to the Longitudinal Direction of the Steel Bars Used in the Experimental Studies.

Although the transgranular character of the brittle portion of the fracture is similar in the martensites that were tempered in the range of 800 to 1200 F, nevertheless the structural features of the tempered martensite become more easily distinguishable as the tempering temperature is raised. This permits observation of the relation of the fracture to the ferrite grain structure of the martensite. For example, at 1200 F, it becomes evident with higher magnification that both the ferrite grain boundaries and the ferrite cleavage play parts in the propagation of brittle fracture in soft-tempered martensite. This is illustrated by Fig. 35. Ferrite cleavage may be seen at arrow 1. Arrow 2 indicates ferrite grain boundaries that participate in the fracture path. These ferrite grain boundaries are not usually the ferrite grain boundaries that correspond to the limits of the prior austenite grains.

At arrow 3 in Fig. 36, transgranular cleavage across a number of ferrite grains is indicated. Similarly-orientated ferrite grains derived from martensite needles are commonly observed to etch alike and thus appear as one large ferrite grain. This feature is frequently observed in the soft tempered martensite.

ANISOTROPY

The orientation of the test specimen with respect to the direction of rolling elongation in rolling or forging affects the ductility values obtained in any given mechanical property test. The relation of ductility to orientation may be seen in the reduction of area and elongation values obtained in the tension test and in the maximum energy values obtained in the V-notch Charpy test.

With the notched bar impact tests, the critical geometrical feature is the position of the notch in the specimen with respect to the axis of elongation during rolling or forging. By convention, a longitudinal Charpy specimen has its long axis parallel with and the notch at right angles to the direction of rolling. There are further conventions for the notch where more than one geometry is allowable, for example, in specimens taken from a flat bar or plate. Flat bars were used in the present study. The methods of sampling and the orientations of the notches in the specimens are illustrated by Fig. 37.

The maximum energy levels obtained in the V-notch Charpy tests have long been known to be consistently higher for longitudinal than for transverse specimens, if both types of specimens are taken from the same piece of steel and have received identical heat treatment. The general trend to be expected may

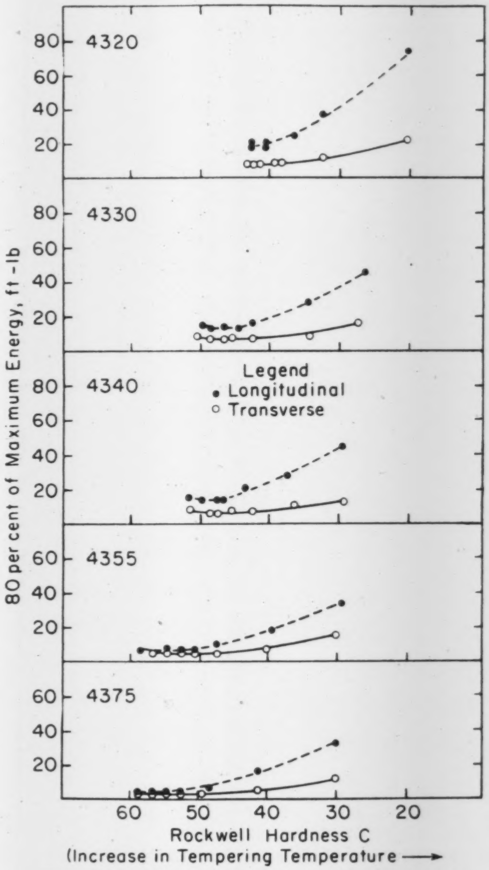


FIG. 38.—Effect of Specimen Orientation on 80 per cent of Maximum Energy Values, from V-Notch Charpy Tests of Tempered Martensites of Different Hardness Levels Produced in 4300 Steels of Various Carbon Contents.

be seen in Fig. 38. In the 4300 steels under consideration, the difference between longitudinal and transverse values varies with the hardness and carbon content. The difference in the absorbed energy is greater the lower the hardness and carbon content.

Longitudinal and transverse properties determined by the V-notch Charpy test give some indication by their divergence of the best and the worst to be expected of a given piece of steel. Neither extreme may be realized in actual service due to the stress conditions present in the machine part and the relation of the stress pattern to the directional properties of the steel. Indeed, it is a rule of good design that the worst directional properties of the steel should be placed in the part in such a way that they are not subjected to the most unfavorable combinations of stress.

The differentials by which the maximum energy values obtained with longitudinal specimens exceed the values obtained with transverse specimens are not always those shown in Fig. 38. The difference may depend upon a number of factors. Among these may be mentioned the segregation structure of the ingot, fortuitous inclusions in the ingot, and, with small reductions, the amount and nature of the reduction process used in fabrications.

The energy curves for transverse specimens were similar to those obtained with longitudinal specimens, except that the maximum energy levels were much lower and the energy transitions less pronounced. In view of the low maximum energy values, determination of the transverse transition temperatures at 80 per cent of the maximum energy was more uncertain than in the case of longitudinal specimens; however, in the 4300 grades the transverse transition temperatures were about the same or lower than the corresponding longitudinal transition temperatures.

While the above conclusion was valid for the 4300 grades and a few others that were investigated in both the transverse and longitudinal directions, data reported in the literature indicate that this relation may not hold universally. Transverse notched bar properties need more study from a metallurgical standpoint.

SUMMARY

1. The effects of carbon and alloy contents on the impact properties of quenched-and-tempered steels can be discussed in terms of their effects on the various types of embrittlement exhibited by tempered martensite, namely, A-brittleness (so called 500 F embrittlement), B-brittleness, and temper brittleness.

All the steels examined exhibit A-brittleness, which is that embrittlement which developed in the tempering range of 500 to 800 F. The amount of A-brittleness and the tempering temperature at which the peak occurs are functions of carbon and alloy content. The amount is greatest for the 0.80 per cent carbon heat in each grade. In the case of 4340 steel the peak occurs at the highest temperature, 800 F, and this grade has the lowest transition temperatures at the low-tempering temperatures, that is, high-strength levels. The straight manganese 1300 grade exhibits the greatest amounts of A-brittleness in general while the molybdenum-bearing grades exhibit the least embrittlement.

B-brittleness, relatively high transition temperatures exhibited at a tempering temperature of 1200 F, is developed in the 2300 series. This is believed to be caused by the presence of austenite formed on tempering, which transforms to martensite on quenching from tempering, or upon testing.

Temper brittleness, developed by tempering in the range from 950 to 1150 F for extended time or by cooling slowly through this range, is observed in the 0.80 per cent carbon heat of most of the grades investigated. Addition of molybdenum minimizes temper brittleness.

No one alloy grade is superior in respect to low transition temperature at all hardnesses and carbon contents. The spread between grades is great at some hardnesses and small at other levels. Generally speaking, the various grades containing molybdenum among them had the lowest transition temperatures at most of the carbon contents and over most of the hardness range.

2. Phosphorus has an adverse effect on the transition temperatures of 4100 and 5100 grades of steel. Molybdenum offsets this adverse effect to a large extent.

3. In general, transition temperature varies directly with austenitic grain size for 2340 and 4140.

4. Metallographic studies correlating impact behavior with the microscopic features of fracturing are presented.

5. For the 4300 grade, maximum absorbed energy is greater for Charpy specimens cut out longitudinally to the working direction than transversely to the working direction, the difference being greater the lower the hardness and carbon content.

Acknowledgment:

The authors are happy to acknowledge the contributions made to this program by the following: I. Kramer, formerly of the Office of Naval Research, and J. Harwood, presently Head, Materials Branch, Office of Naval Research, who provided technical guidance and encouragement during this program, and Mary Baeyertz and W. F. Craig, Jr., formerly of Armour Research Foundation, who were responsible for the supervision of this program during its early stages.

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DISCUSSION

MR. SAMUEL J. ROSENBERG[1].—I should like to comment on the criterion for transition temperature that has been used in the paper. The criterion used was 80 per cent of the maximum energy. This raises the question as to just what do the authors consider the maximum energy to be. Is it the value at room temperature? Suppose higher values were obtained at lower or higher temperatures, would the highest value so obtained be used?

The authors say that this criterion corresponded approximately to the onset of brittle fracture. This statement may be applied only to steels having a completely fibrous fracture at the temperature used for selecting the maximum value. Many steels, either because of heat treatment or of composition, do not have a fibrous fracture at the highest test temperature used so that taking 80 per cent of the maximum energy value is meaningless as a criterion for transition temperature of such steels in the sense used by the authors. In the curves shown toward the latter part of the presentation, where energy values ranged roughly from about 15 to 30 ft-lb, I would guess that even at room temperature the steels were predominately brittle in their fracture characteristics.

I should like to suggest that the task of setting a standard definition for transition temperature is a suitable job for the American Society for Testing Materials.

MR. OSCAR O. MILLER.[2]—There has been a tremendous amount of work done here, and it is very useful and interesting. Having had the opportunity of seeing some of the initial reports that were written, this publication is a synopsis of all those reports.

A large number of heats were investigated. There is one thing missing that should be considered for further work, and that is the absence in any instance of two heats of the same general type, so that we do not know the variability between heats. This could be very important, because it gives a basis on which to judge how significant the results may be.

Another thing is, there is straight manganese, straight chromium, and straight nickel steel here, but no straight molybdenum steel. Of course this may be because there is no straight molybdenum steel of this type being used, but in speaking of the individual effect of alloying elements something on molybdenum would be very useful.

MR. GEORGE SACHS.[3]—I have been working on super-high strength steels for many years and developed the notch-tension test for the purpose of evaluating the tendency of such steels for embrittlement in the presence of stress concentrations. This test is now also extensively used for a variety of other materials, including high-temperature alloys, titanium, and ship steels.

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[2] Head, Steel Section, The International Nickel Co., Inc., Bayonne, N. J.

[3] Director, Metallurgical Research Lab., Syracuse University, Syracuse, N. Y.

Recently, there has been an opportunity to compare the scattering of impact-test and notch-tension data, presented in the accompanying Fig. 39. The impact energy is found to scatter considerably more than the notch strength. This means that a large number of parallel tests would be required to arrive at accurate impact data. In addition, the transition of heat-treated steels is not sudden within a narrow temperature range, as that of normalized steels, but very gradual. It appears, therefore, rather difficult to establish a definite transition temperature for such steels from impact data. The above-shown graph illustrates frequency curves. To obtain such an expanded set of test values the following procedure was used: (1) Each set of test data was averaged and the average value was assigned "zero" deviation, (2) the percentage deviation from this value was determined for each test of the set, (3) such results of a number of sets, differing in heat treatment, were plotted in the same coordinate system.

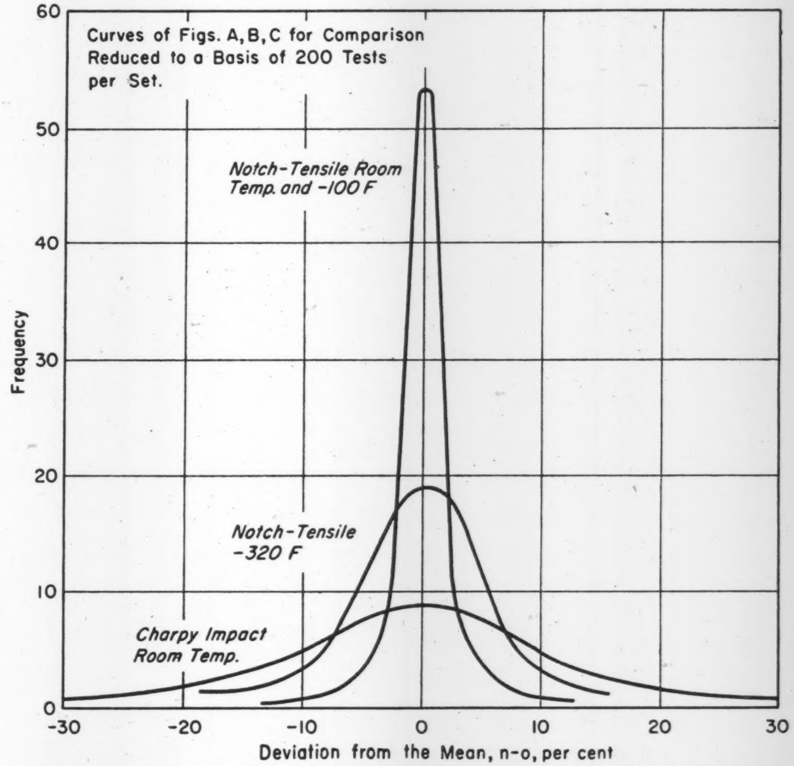

FIG. 39.—Comparison of Distribution Curves for Respective Sets of Data.

MR. DAVID E. DRISCOLL[4] (*by letter*).—The authors are to be congratulated on the thoroughness used in their investigations. With one exception, the statements are ably supported by data. The one exception is the effects of rare earth on 4330 steels containing sulfur. The authors infer that the addition of rare earth increases the energy values by a ratio of approximately 4 to 1. Figures 37 and 38 show that rare earth additions to 4330 steels having 0.020 and 0.038 per cent sulfur have no effect on longitudinal impact properties until tempering temperatures of 1000 F are reached. Even then the effects are small, but these same rare earth additions do have an effect on the transverse properties of both the 0.020 and 0.038 per cent sulfur bearing steels. In this case, the energy values are increased by an approximate ratio of 2 to 1.

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[4] Chief, Mechanical Test Section, Watertown Arsenal, Watertown, Mass.

MESSRS. J. P. SHEEHAN AND HARRY SCHWARTZBART (*authors' closure*).—In answer to Mr. Rosenberg's questions concerning the use of 80 per cent of maximum energy as a criterion for transition temperature, we offer the following explanation. For all steels tested, it was found that the impact energy testing temperature curve reached a maximum with increasing temperature and then remained fairly constant with further increase in testing temperature. This was true for all conditions of heat treatment and hardness. With the softer tempered steels there was an accompanying change in the fracture appearance from cleavage to shear, and completely shear fractures were obtained at about 80 per cent of maximum energy. The higher hardness steels did not undergo a clear-cut change in fracture appearance and in many cases remained partially brittle even when the testing temperature was equivalent to the tempering temperature, that is 400 to 700 F. Thus, it was impossible to select a fracture criterion of transition that would apply to all conditions of heat treatment except for one at the lower end of the transition curve where the fractures were predominantly brittle. Therefore the 80 per cent of maximum energy criterion, which could be applied to the steels at all hardness levels, was chosen.

With regard to Mr. Miller's comments, we agree that it would have been very interesting to test several heats of the same grade to determine the variability between heats. Such a project is currently being conducted on ship plate steels by the National Bureau of Standards under the joint sponsorship of the Ship Steel Committee and American Iron and Steel Institute. Also, data on a straight molybdenum grade would have been interesting but perhaps not useful since these steels are not used in large quantities.

The influence of rare earths on transverse properties at the higher tempering temperatures was found to be quite beneficial. As Mr. Driscoll points out, there was an increase in maximum impact energy of about 100 per cent with rare earth additions. Our reference to a ratio of 4:1 may have been misleading. We were merely citing the difference between the high (0.038 per cent) sulfur steel without rare earth additions (about 15 ft-lb) and the low (0.020 per cent) sulfur steel with rare earth additions (about 60 ft-lb), which is in the ratio of 1:4.

CRACK INITIATION AND PROPAGATION IN THE V-NOTCH CHARPY IMPACT SPECIMEN*†

By Carl E. Hartbower[1]

Synopsis

Because the energy value obtained from conventional V-notch Charpy impact tests integrates the crack-initiation and crack-propagation stages of the fracture process, it was deemed necessary to investigate a modification of conventional impact testing technique to permit differentiation between the crack initiation and propagation stages.

Two approaches were investigated. Both involved the initiation of a crack by means of an initial low-energy blow of the impact-machine pendulum. The first method determined the highest temperature at which the initial low blow caused complete fracture of the test specimen, that is, the temperature at which the low blow initiated a crack which then propagated by continuous release of the elastic-stress field developed by the initial low blow. The second method required the measurement of lateral expansion after the crack-initiating low blow and again after a second, fracturing blow.

Multiple low blows delivered at or below the brittle-fracture transition temperature provided a possible explanation for brittle fractures in service which have occurred under conditions of static load or low nominal stress.

Previous Work:

This report is the third of a series with the over-all objective of evaluation and improvement of the Charpy impact test. The first paper discussed the Poisson effect as it occurs in Charpy impact specimens (**1**).[2] It demonstrated that the deformation attending the fracture of a Charpy bar can be measured as either lateral contraction occurring under the notch or lateral expansion at the compression surface of the test bar. In pearlitic steel, a linear relationship was shown to exist over an appreciable range of energy absorption between lateral contraction, lateral expansion, and energy to fracture.

The objective of the second paper was to evaluate certain performance criteria and a particular definition of transition temperature for use in V-notch Charpy *slow-bend* tests (**2**). Transition temperature was defined as the highest temperature at which sudden and complete fracture occurred at maximum load, that is, zero energy after maximum load (Fig. 1). The definition was based on (*a*) an experimental observation by Raring (**3**) that the start of crack propagation in the

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[*] Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1957.

[†] The opinions or assertions contained herein are the private ones of the author and are not to be construed as official or reflecting the views of the Dept. of the Army.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 538.

thickness direction of V-notch Charpy slow-bend bars coincides with the maximum in load as determined from the load-deflection diagram and (*b*) a suggestion by Orowan (4) that one may differentiate between ductile and brittle fracture by noting the manner in which the initiating crack is propagated—a brittle fracture crack is propagated by continuous release of the elastic stress field surrounding the crack, whereas a ductile fracture crack requires plastic-deformation work to extend the crack. Thus, with zero energy (work) after maximum load, fracture was considered to be brittle. The deformation (lateral expansion) measured in the specimens which suddenly and completely fractured at maximum load was assumed to have occurred prior to crack propagation. An appreciable amount of deformation was found to occur in V-notch Charpy slow-bend specimens at the transition temperature; it was concluded that the amount of deformation attending brittle fracture provided a measure of the relative embrittlement of a series of steels or a given steel under various metallurgical conditions. Based on the assumption that the deformation measured in the fractured test specimen preceded brittle crack propagation, it was concluded that the deformation measured in specimens developing brittle fracture reflected only the crack-initiation stage of fracture, that is, the resistance of the material to bending and formation of the initiating crack.

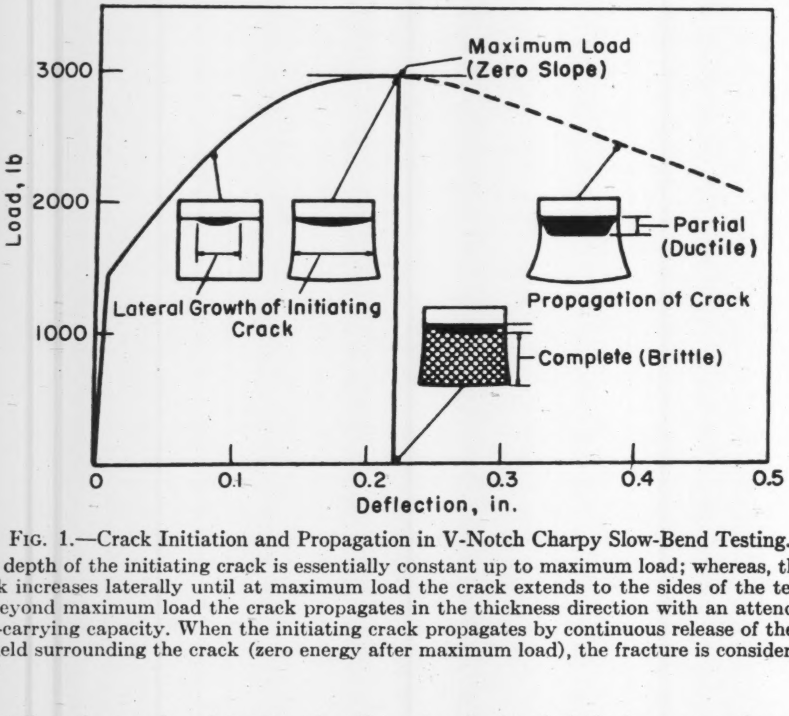

FIG. 1.—Crack Initiation and Propagation in V-Notch Charpy Slow-Bend Testing.

The depth of the initiating crack is essentially constant up to maximum load; whereas, the width of crack increases laterally until at maximum load the crack extends to the sides of the test specimen. Beyond maximum load the crack propagates in the thickness direction with an attendant loss of load-carrying capacity. When the initiating crack propagates by continuous release of the elastic-stress field surrounding the crack (zero energy after maximum load), the fracture is considered to be brittle.

In V-notch Charpy impact testing, the energy value read from the calibrated machine pendulum constitutes an integration of the various stages of the fracture process, that is, a summation of the energies required for bending, crack initiation, and crack propagation. A technique used at the Naval Research

Laboratory (NRL) (5) provides a method for separating the crack initiation and crack propagation stages. In the work conducted at NRL, specimens were tested in an impact machine in the conventional manner except that the kinetic energy of the hammer was varied by changing the mass or velocity of the hammer. Using multiple low-energy impact blows on medium-carbon steels, the investigators reported that the energy required to initiate a crack is constant over a wide range of temperature, whereas the energy absorbed in extending a crack varies with temperature (Fig. 2).

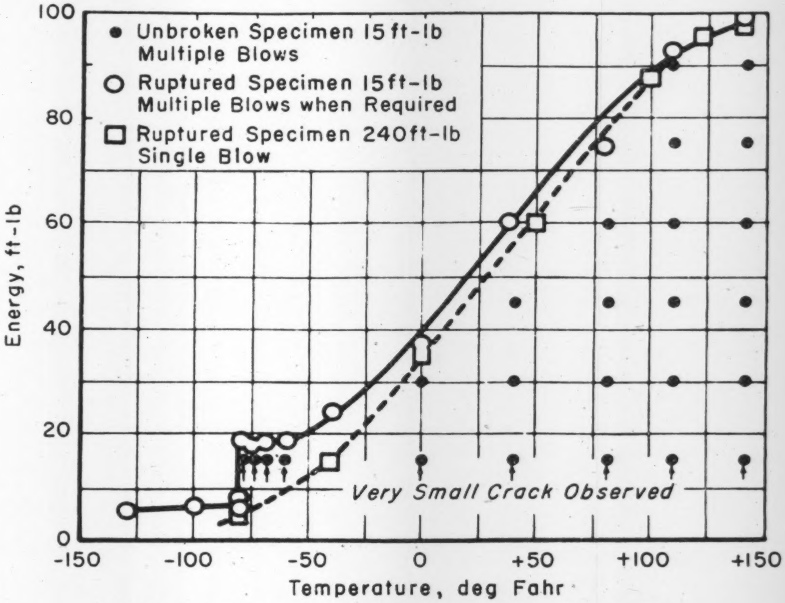

FIG. 2.—Transition Behavior Obtained from Low-Energy Blows.

OBJECTIVE

It is the objective of this investigation to introduce a simple variation in the NRL low-blow technique which will permit differentiation between the crack initiation and crack propagation stages of fracture in V-notch Charpy impact testing.

MATERIALS

The material used in this investigation was a single heat of steel purchased in the form of $\frac{9}{16}$-in. square, annealed, machine-straightened, aircraft quality, electric furnace AISI 4340 hot-rolled steel rods.

The chemical analysis obtained from 20 samples picked at random from the rods was as follows:

Carbon, per cent	0.37 to 0.38
Manganese, per cent	0.75 to 0.77
Silicon, per cent	0.27 to 0.28
Sulfur, per cent	0.013 to 0.015
Phosphorus, per cent	0.013 to 0.016
Nickel, per cent	1.73 to 1.77
Chromium, per cent	0.83 to 0.86
Molybdenum, per cent	0.25 to 0.27

Three heat treatments were used to develop a variation in hardness (strength) in the AISI 4340 steel:

Condition A (Rockwell Hardness C 27)

1. Heated to 1600 F, held 1 hr, oil quenched

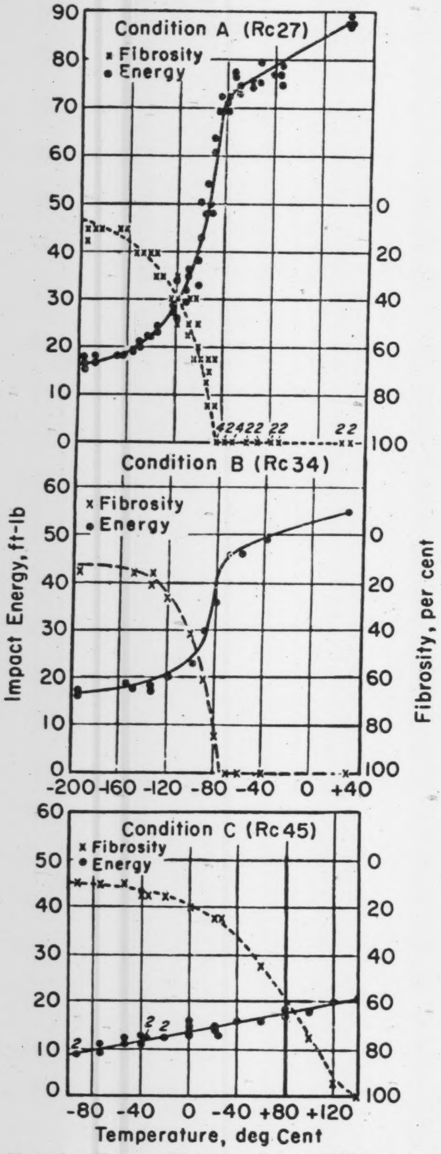

FIG. 3.—Energy-Temperature and Fibrosity-Temperature Transition Curves as Obtained by Conventional V-Notch Charpy Impact Tests of Material—Conditions A, B, and C.

2. Heated to 1200 F, held 1½ hr, oil quenched
3. Heated to 570 F, held 3 hr, air cooled

Condition B (Rockwell Hardness C 34)

1. Heated to 1600 F, held 1 hr, oil quenched
2. Heated to 1030 F, held 1½ hr, oil quenched
3. Heated to 570 F, held 3 hr, air cooled

Condition C (Rockwell Hardness C 45)

1. Preheat to 1150 F
2. Heated to 1550 F (bumper furnace), held for 15 min, oil quenched
3. Packed in dry ice for 10 to 12 hr
4. Heated to 780 F, held 2 hr, oil quenched
5. Heated to 375 F, held 3 hr, air cooled

The microstructure produced by the above heat treatments was as follows:

Condition A (*Rc 27*)—tempered martensite with considerable fine carbide present and some evidence of grain boundary carbide.

Condition B (*Rc 34*)—tempered martensite with only a small amount of carbide present, mostly around the grain boundaries; the structure showed evidence of acicularity.

Condition C (*Rc 45*)—tempered martensite with only a trace of fine carbide; the structure was acicular.

Figure 3 presents the transition curves as obtained by conventional testing procedure for material conditions A, B, and C.[3] From these curves it may be noted that there is very little difference indicated between conditions A and B except in the energy level corresponding to the upper portions of the energy-temperature curves. Condition C, on the other hand, is indicated to be relatively notch-sensitive with no abrupt transition from high to low energy values.

TEST METHOD

The proposed variation in low-blow technique consisted of three steps: First, the energy to initiate cracking was determined; second, the effect of temperature (in the ductile fracture range) on the incidence and extent of initial cracking was

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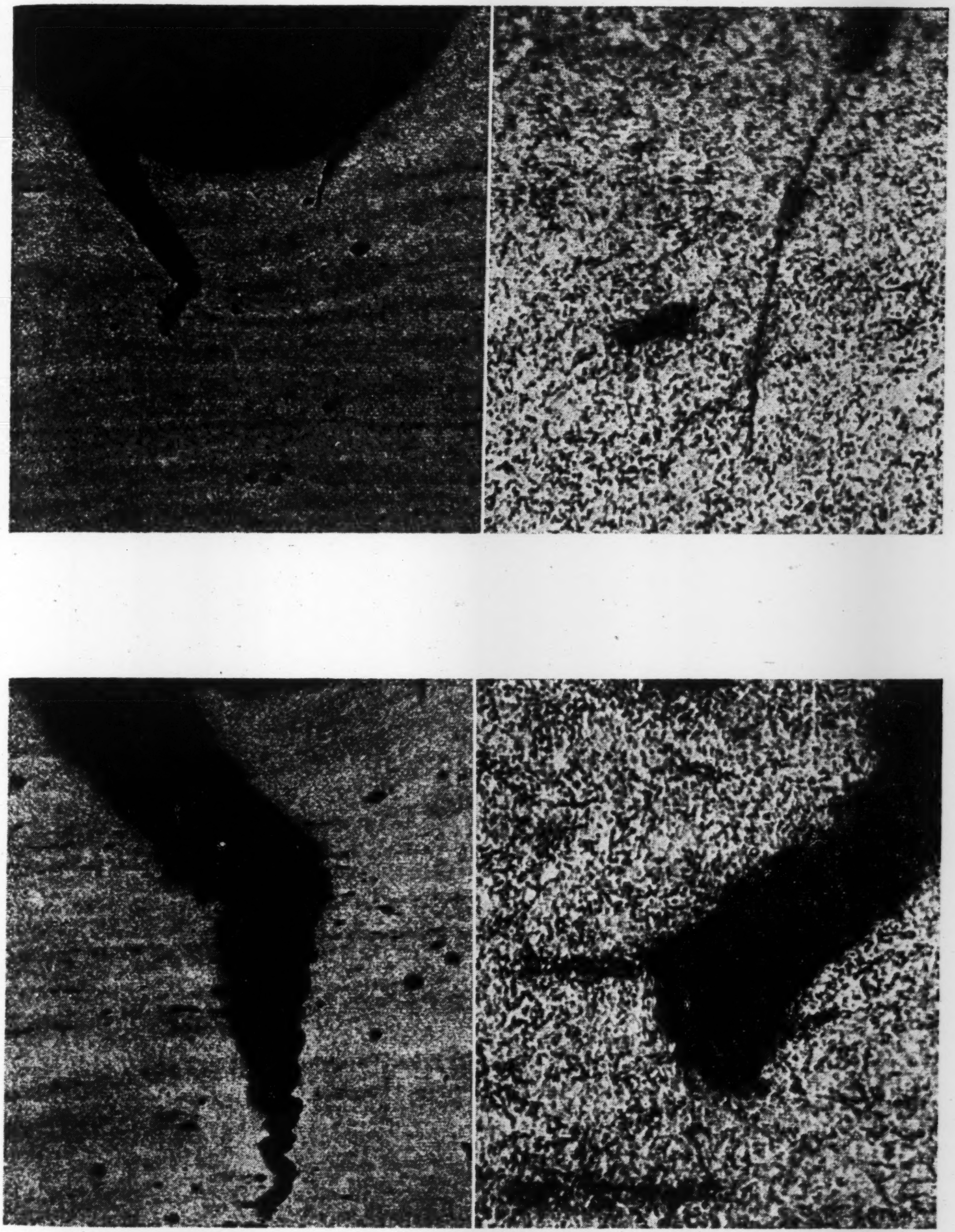
[3] Data supplied through the courtesy of Messrs. T. S. DeSisto and D. E. Driscoll of the Mechanical Metallurgy Branch, Watertown Arsenal Laboratories.



×100 ×1000

FIG. 4.—Initiating Cracks in Material—Condition A (Rc 27).

The upper photomicrographs show typical cracks produced by a 20 ft-lb low blow at −100 C; the lower, by a 30 ft-lb low blow at −100 C.

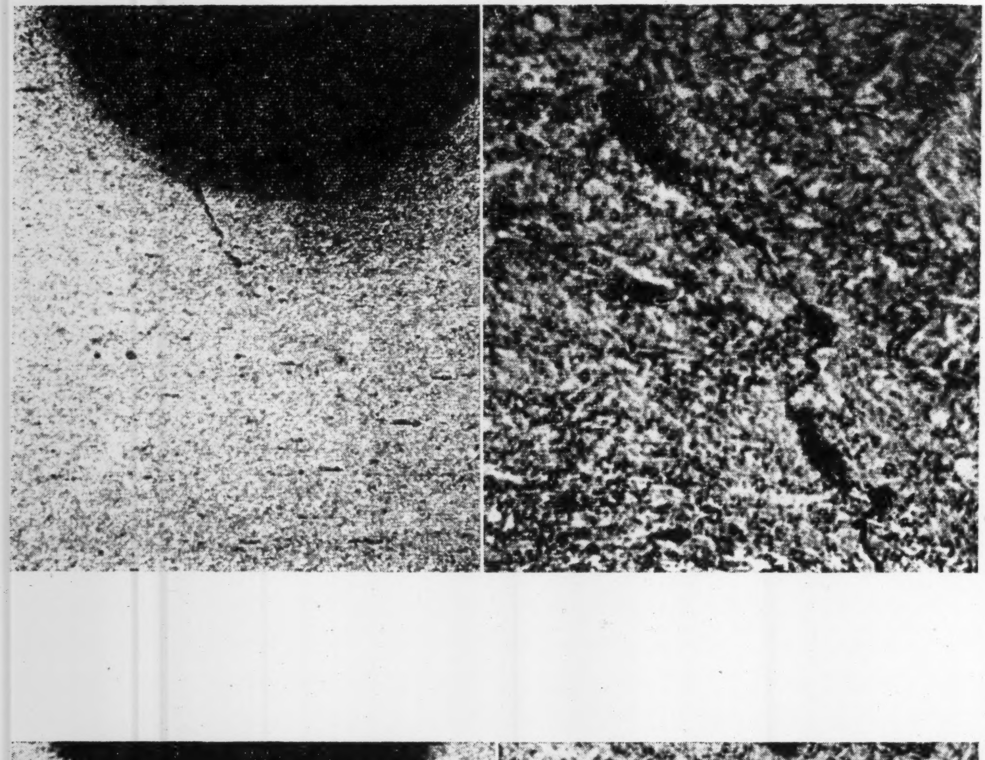


FIG. 5.—Initiating Cracks in Material—Condition B (Rc 34).

The upper photomicrographs show typical cracks produced by a 15 ft-lb low blow at −100 C; the lower, by a 20 ft-lb low blow at −100 C.

investigated; and, third, the onset of brittle fracture was determined by noting the highest temperature at which either the crack-initiating low blow resulted in complete fracture or the low-blow-initiated crack propagated under a second blow without evidence of plastic deformation work.

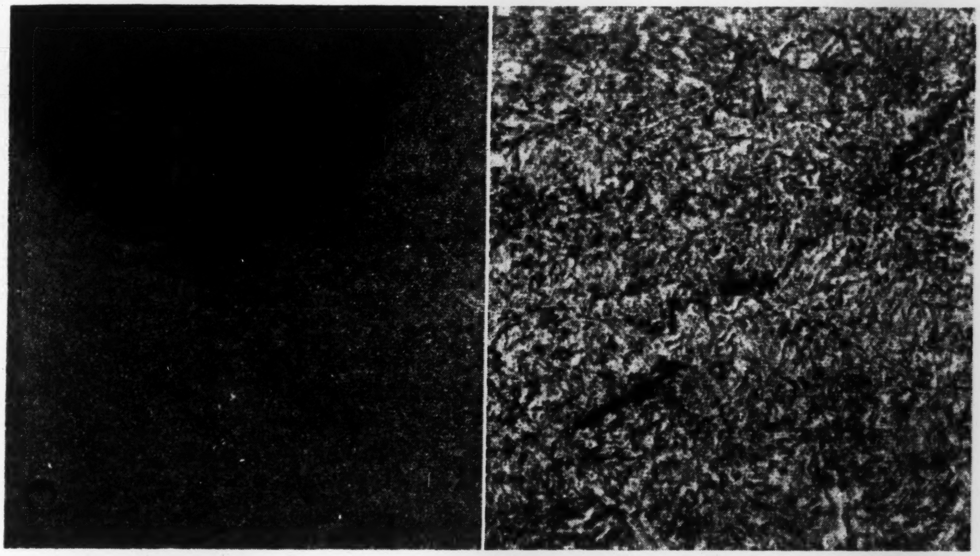

× 100 × 1000

FIG. 6.—Initiating Shear Crack Produced by a 12 ft-lb Low Blow in Material-Condition C (Rc 45) at Room Temperature.

To determine the crack-initiating energy level, specimens were delivered a variety of low blows with one blow to a specimen. After delivering a low blow, a drop of India ink containing a wetting agent (Kodak Photo-Flow solution) was deposited in the Charpy V-notch of those specimens which did not fracture under the initial blow. By action of the wetting agent and capillarity, the ink flowed into the crack, if present, staining the crack surfaces black. The test specimens were then fractured by means of a full blow of the impact-machine pendulum. Examination of the fracture surfaces for the presence of staining established the crack-initiating energy level for the materials under investigation.

Attention is invited to the fact that the distinction between the crack-initiation and crack-propagation stages of the fracture process was less precise than the author would have liked. This was dictated by the practical difficulty of distinguishing between the various stages of the crack initiation process. It was found (Figs. 4, 5, and 6) that in the first stage, the initiating crack consisted of shear with so little separation of the fracture surfaces that detection by capillary ink staining was not reproducible.[4] In the second stage of cracking, the initiating crack changed direction, extending perpendicular to the principal stress. At this stage, ink staining provided an easy and reproducible method of crack detection. Therefore, for purposes of this investigation, the term "initiating crack" refers to a low-blow induced crack which can be detected by ink staining.

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[4] Even metallographic examination sometimes failed to disclose an extremely fine initiating shear crack which could be detected only after an external load was applied to the metallographic specimen while being viewed on the microscope (S. V. Arnold, unpublished study).

After the crack-initiating low blow, specimens were then tested with a full-energy blow over a wide range of temperature producing ductile fracture. To control the extent of possible strain aging, the time between delivering the low blow and subsequent fracturing of the test specimen was held to 24 hr ±8 hr. Both the low blow and final blow were delivered at the same temperature. The effect of testing temperature on the incidence and extent of low-blow cracking was noted by observing the ink staining on the crack surfaces after such specimens were fractured. In addition to the presence of cracking, the following data were recorded:

1. Magnitude of low blow,[5]
2. Temperature of test specimen,
3. Lateral expansion produced by the low blow,
4. Lateral expansion in the fractured specimen,[6]
5. Energy absorbed in the fracturing blow.

To resolve questions on the effect of deflection velocity, a smaller hammer was attached to the impact machine pendulum (15-lb in lieu of the standard 60-lb hammer). Thus, at a given energy level, the smaller hammer raised to a greater height provided a higher hammer velocity. Tests were also conducted using a drop weight machine, which made it possible to evaluate the crack-initiating energy level over a somewhat wider range of deflection velocities.

DISCUSSION OF RESULTS

The Initiating Crack:

In all three material conditions (R_C 27, 34, and 45), initial cracking appeared to occur as a result of the shear stress at the free surface of the V-notch (Figs. 4, 5, and 6). Cracking was observed to occur in two stages. Initially, the shear crack occurred at approximately 45 deg to the center line and at either side of the apex of the machined notch, that is, on a trajectory of shear stress. After progressing a short distance, the shear crack changed direction to propagate normal to longitudinal stress. The extension of the shear crack in a direction perpendicular to the principal stress is the second stage of the fracture process. These two stages of cracking are hereinafter referred to as the shear and fibrous portions of the initiating crack. Except for direction, there was very little difference between the shear and fibrous portions of the initiating crack when viewed in cross-section under the microscope. The shear crack in fact had the appearance of discontinuous cleavage at high magnification (Figs. 4, 5, and 6) and in some instances very much resembled portions of the fibrous crack. However, when viewed in the fracture surface of a broken test specimen, the two portions of the initiating crack were easily distinguished because of the notably smooth appearance of the shear. In material conditions A and B, both the shear and fibrous portions of the initiating crack were distinctly visible in the fracture surface of broken test specimens at all temperatures down to and including the brittle fracture transition temperature (Fig. 7). In condition C, on the other hand, the fibrous portion of the initiating crack disappeared at low testing temperatures, leaving only the shear portion in evidence.

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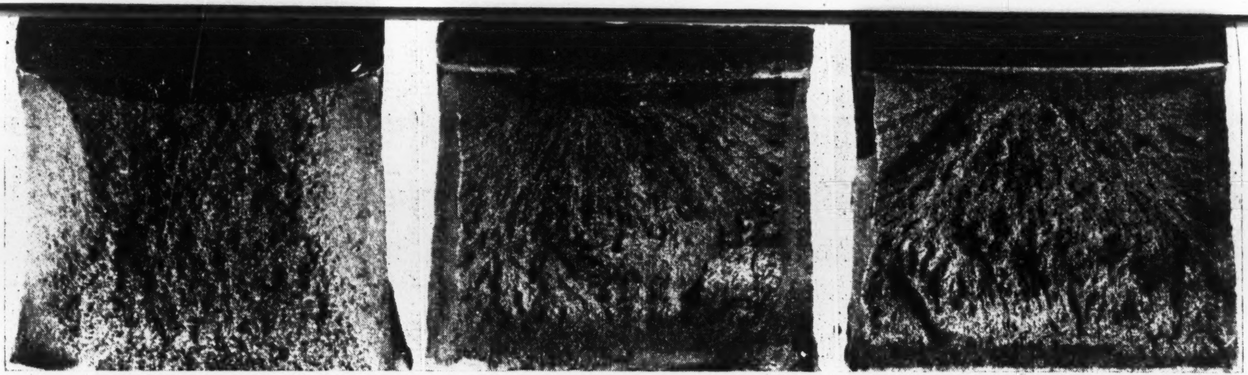
[5] When the low blow was sufficient to cause fracture, the value of the energy absorbed in fracturing the specimen was recorded.

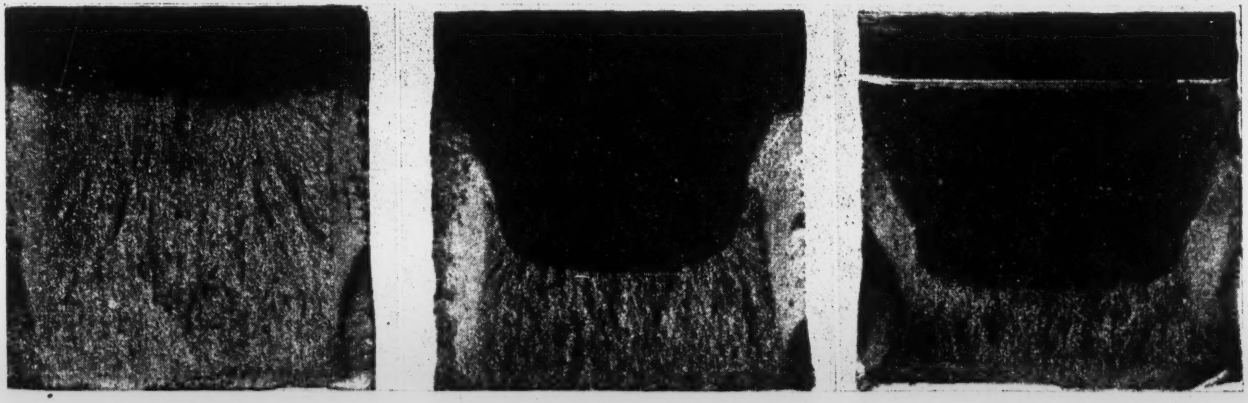
[6] By subtracting the lateral expansion produced by the low blow from the total expansion occurring in the fractured test specimen, a measure of the plastic deformation work required to propagate the initiating crack was determined.



−80 C −120 C −140 C

FIG. 7.—Extent of Cracking as a Function of Temperature.

The depth of the initiating crack produced by a given low blow (30 ft-lb in this illustration) was found to be essentially constant over a wide range of temperature. At −80 and −120 C the 30 ft-lb blow resulted in ink-stained cracks as illustrated. The cracking shown for −80 C (100 per cent fibrous fracture) is typical for all higher temperatures tested. The −140 C fracture occurred as a result of the initial blow with the initiating fibrous crack approximately the same depth as ink-stained cracks at −80 and −120 C.



13 ft-lb 14 ft-lb 15 ft-lb

FIG. 8.—Extent of Cracking as a Function of Energy in Material-Condition C (Rc 45).

Unlike material-conditions A and B, a small increase in the energy of low blow caused a marked increase in the depth of the low-blow induced crack (+120 C).

Because of the extreme fineness and sometimes discontinuous nature of the shear crack, detection by means of ink staining at the first stage of crack initiation was found to be uncertain. However, once the initiating crack changed direction and became fibrous, ink staining was found to be both a reproducible and expeditious method of crack detection.

The Crack-Initiating Energy Level:

In the case of the material in condition A (R_C 27), a low blow of 15 or 20 ft-lb produced no cracking as determined by staining. With a 25 ft-lb blow, however, stained shear cracks developed in some specimens at the apex of the V notch. At 30 and 40 ft-lb, cracks were a combination of shear and fibrous fracture. The extent of the initiating fibrous crack as produced by a given low-blow energy level was found to be largely independent of temperature over a wide range (see Fig. 7).

In the case of condition B (R_C 34), a room temperature low blow of 15 ft-lb produced no cracking as determined by staining. With a 20 ft-lb blow, however, a stained fibrous crack was observed. At 25 and 30 ft-lb, cracking became progressively deeper. Again the extent of the initiating fibrous crack was found to be largely independent of temperature over a wide range.

In the case of condition C (R_C 45), a room-temperature low blow of 10 ft-lb produced no cracking as determined by staining. With a 12.5 ft-lb blow, a stained shear crack was observed. With a 15 ft-lb blow, specimens fractured with an energy absorption of 13.5 ft-lb. Subsequent tests to determine the crack-initiating energy level were conducted at +120 C. At this temperature, a 13 ft-lb low blow was found to produce a stained shear crack (Fig. 8). Low blows of 14 and 15 ft-lb, on the other hand, caused a marked increase in the depth of the low-blow-induced crack.

In all three material conditions, the initial cracking occurred on a shear-stress trajectory, progressed for a short distance under the notch and then changed direction under action of the principal tensile stress to form a fibrous crack. Based on the presence of a fibrous crack as defined heretofore, the approximate crack-initiating energy levels (and attendant deflection rates) were as follows:

Material Condition	Energy, ft-lb	Velocity, ft per sec
A (R_C 27)	30	5.7
B (R_C 34)	20	4.6
C (R_C 45)	13	3.8

Effect of Velocity:

Because the low-blow technique introduces a variable deflection rate, a check was made as to whether there was a velocity effect in the case of material conditions A and B. Therefore, the 60-lb pendulum hammer was replaced with a 15-lb hammer; thus, for a given low-energy blow, the 15-lb hammer, raised to a greater height, increased the striking velocity by a factor of two. In condition A (R_C 27), the energy level corresponding to initiation of a fibrous crack was the same for both hammers (30 ft-lb at 5.7 ft per sec with the 60-lb hammer and 30 ft-lb at 11.4 ft per sec with the 15-lb hammer). Results in condition B (R_C 34) using the 15-lb hammer were anomalous in that a slightly greater energy was required to initiate fibrous cracking with a 15-lb hammer (25 ft-lb at 10.4 ft per sec) than with a 60-lb hammer (20 ft-lb at 4.6 ft per sec). The expected effect (if any) of increased deflection rate was to produce cracking at a lower energy level. To resolve the anomalous behavior in condition B and to obtain a wider range of velocity, drop-weight tests were conducted. The results confirmed the crack-initiation energy level obtained with the standard 60-lb hammer. Moreover, variations in drop-weight velocity between 4 and 16 ft per sec had no effect on the energy required to initiate cracking. The anomalous behavior with the 15-lb hammer was attributed to vibration losses (see note).

NOTE.—In a study of the influence of pendulum flexibilities on impact-energy measurements, Bluhm has shown that energy lost by vibration in the impact machine pendulum can give rise to a significant error in impact test results (6). The study was prompted by the observation that material condition C (Rc 45) was found to fracture with remarkable and unfailing reproducibility in one pendulum-type machine having a capacity of 217 ft-lb (16.8 ft per sec) registering an energy absorption of 13 ft-lb and, yet, with the same reproducibility, fail to break in a second machine having a capacity of 16 ft-lb (11 ft per sec).

To demonstrate the absence of a significant strain-rate effect, a series of impact tests were conducted using a drop-weight type machine. The rigid masses were machined to weights corresponding to those of the pendulum machines of 16 and 217 ft-lb capacities. The weights were dropped from heights to provide the same energy and impact velocity levels as the pendulum machines. Both drop-weights (217 ft-lb capacity at 16.8 ft per sec and 16 ft-lb capacity at 11 ft per sec) broke the test specimens (at room temperature) when the weight elevation was such as to provide 13 ft-lb of available energy, but did not fracture the specimen when the weight elevation corresponded to 12 ft-lb. From these tests it was proposed that variations in impact velocity in the range investigated had little or no effect and that flexibility or "softness" of the 16 ft-lb capacity impact machine was the primary factor in producing the anomalous effect.

Effect of a Crack on Transition Behavior:

A previous investigation by Zeno and Low (7) compared the performance of standard V-notched Charpy specimens with Charpys containing a shallow fatigue crack at the bottom of the V-notch. The latter were designated by the investigators as V-notch Charpy bars with an "infinitely" sharp notch. The sharp notch was introduced by subjecting finish-machined V-notch Charpy bars to constant-force fatigue until a shallow fatigue crack of controlled depth (0.010 to 0.019 in.) was produced at the apex of the machined V-notch. Transition curves were obtained by standard testing procedures. Comparing the relative positions of the transition

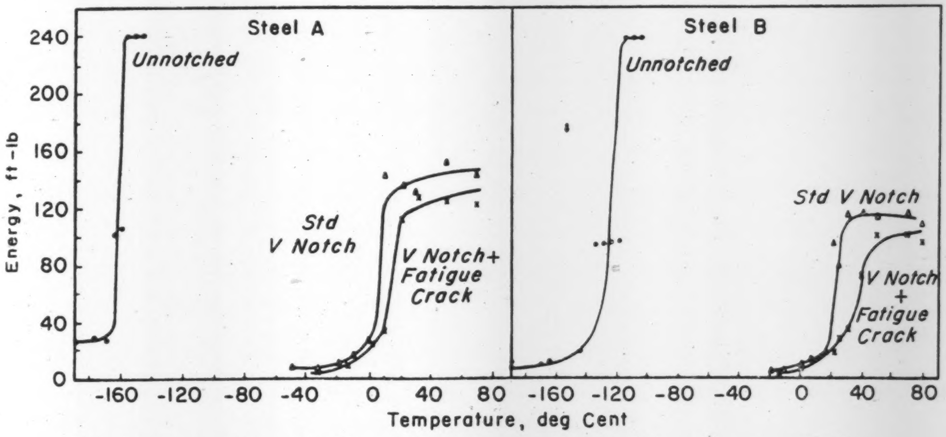


FIG. 9.—Energy-Transition Curves From Average Impact Values (Zeno and Low).

The presence of a natural crack at the apex of the machined Charpy V-notch is shown to have little effect on the transition curve.

curves for standard V-notch Charpys and V-notch Charpy bars with a fatigue crack (Fig. 9) lead to the conclusion that the standard V-notch Charpy specimen approaches very closely the most severe conditions of strain rate and notch severity which may be achieved in this size specimen.

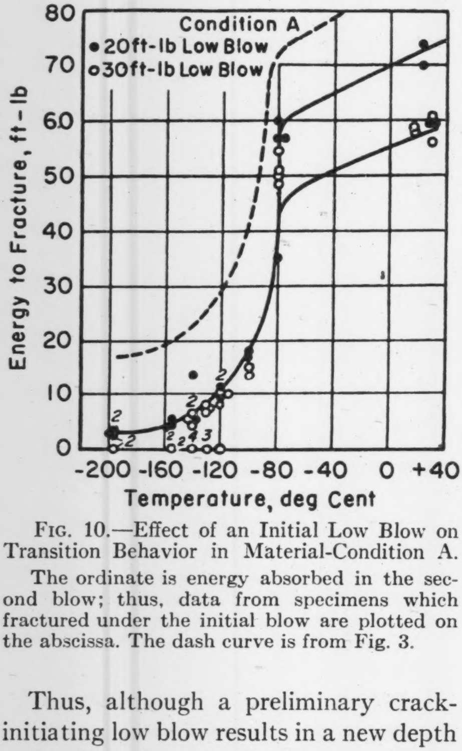


FIG. 10.—Effect of an Initial Low Blow on Transition Behavior in Material-Condition A.

The ordinate is energy absorbed in the second blow; thus, data from specimens which fractured under the initial blow are plotted on the abscissa. The dash curve is from Fig. 3.

Thus, although a preliminary crack-initiating low blow results in a new depth and radius of notch, the findings of Zeno and Low (7) suggest that transition behavior as indicated by energy-temperature transition curves may not be materially affected.

Effect of a Crack-Initiating Blow on Transition Behavior:

In an earlier study of transition behavior in V-notch Charpy *slow-bend-tests* (2), brittle fracture was distinguished from ductile fracture by means of a self-propagating-crack concept. Transition temperature in slow bend tests was defined as the highest temperature resulting in sudden and complete fracture at maximum load (the point on the load-deflection diagram at which the initiating crack, having extended laterally to the sides of the test specimen, propagated in the thickness direction). A crack which propagated suddenly and continuously at maximum load was referred to as self-propagating, that is, it propagated under action of elastic-stress energy alone rather than as a result of plastic-deformation work.

It was the intent of the tests reported herein to apply the self-propagating crack concept to V-notch impact testing by noting the highest temperature at which specimens failed under a crack-initiating low blow. In view of the fact that the low blow consisted of the minimum energy required to initiate (ink-stained) cracking, it is hypothesized that when fracture occurred under the initial blow, the energy of the low blow was largely dissipated as plastic-deformation work necessary to initiation of the crack, and therefore propagation of the crack was accomplished by action of the elastic-stress energy surrounding the crack.

Figure 10 presents the energy-temperature transition curve for material condition A specimens prestrained with 20 and 30 ft-lb blows using both 15- and 60-lb hammers. The energy-to-fracture plotted in the ordinate is the energy absorbed in the second full-capacity blow. Thus, when the specimens fractured under the first blow, the data were plotted on the abscissa, indicating zero energy-to-fracture for a second blow. At temperatures producing crystallinity in the fracture (below -80 C), with the exception of the data from specimens which fractured under an initial low

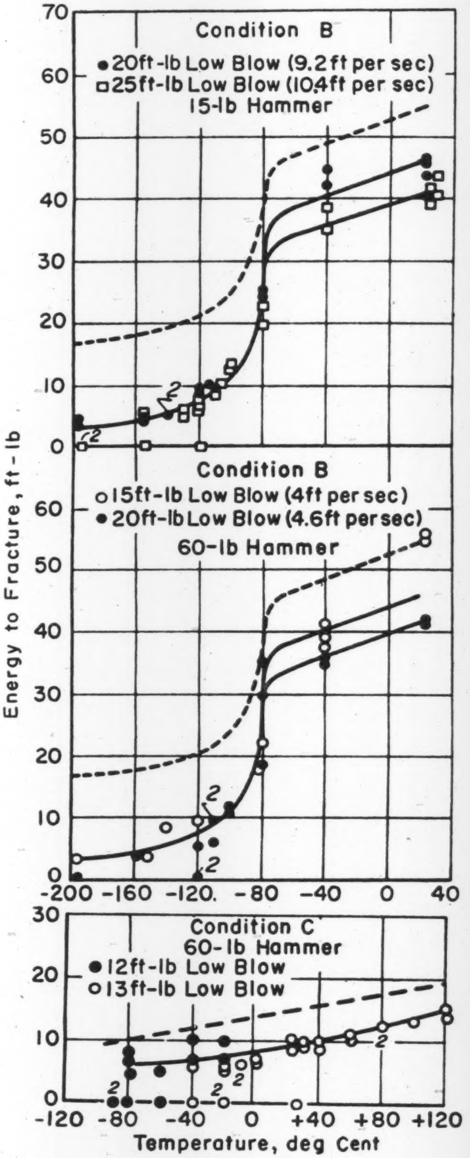


FIG. 11.—Effect of an Initial Low Blow on Transition Behavior in Material-Conditions B and C.

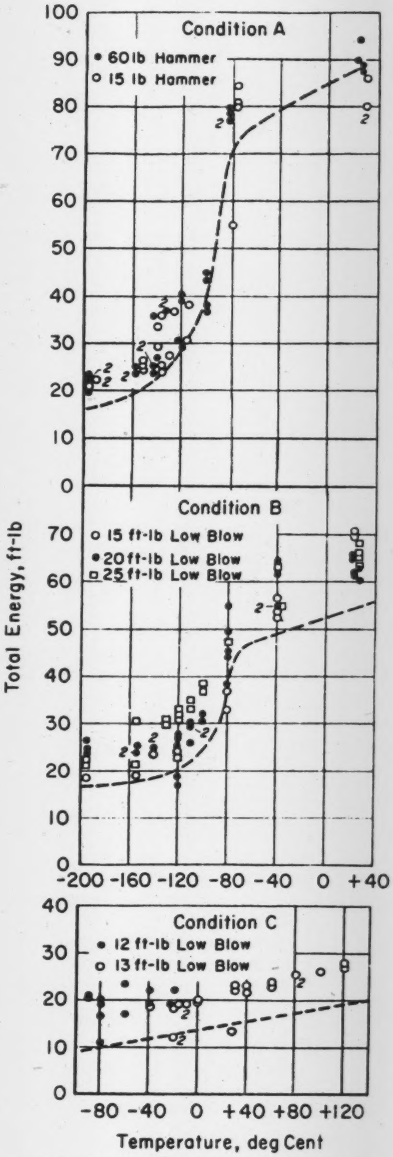


FIG. 12.—Transition Behavior in Material-Conditions A, B, and C as Indicated by the Sum of the Low-Blow Energy and the Energy-to-Fracture.

blow, energy-to-fracture values fell on a single curve. A similar behavior was noted in the case of conditions B and C (Fig. 11). The highest temperatures permitting the formation of a self-propagating crack were as follows:

Condition	Temperature, deg Cent
A (Rc 27)	−120
B (Rc 34)	−120
C (Rc 45)	25

The fact that low energy blows were absorbed without causing fracture at temperatures at which conventional full-capacity blows produced fracture points to a possible defect in the low-blow technique when energy is used as a performance criterion. For example, condition A did not fracture at temperatures down to −196 C when subjected to a 20 ft-lb blow, whereas the conventional transition curve indicates that at −196 C fracture should have occurred at approximately 17 ft-lb. Moreover, the conventional transition curve indicates that at −140 C a 22 ft-lb blow should have been sufficient to cause fracture, whereas four out of the eight specimens delivered a 30 ft-lb blow at −140 C did not fracture. Similar behavior was observed in the case of conditions B and C (Fig. 11). These discrepancies are attributed to the fact that certain portions of the kinetic energy in the pendulum were not effective in producing fracture, such as the elastic-stress energy developed in the specimen at the points of support and in the anvil and hammer upon impact, the energy lost as a result of brinelling of the test specimen, rebound of the pendulum, vibration in

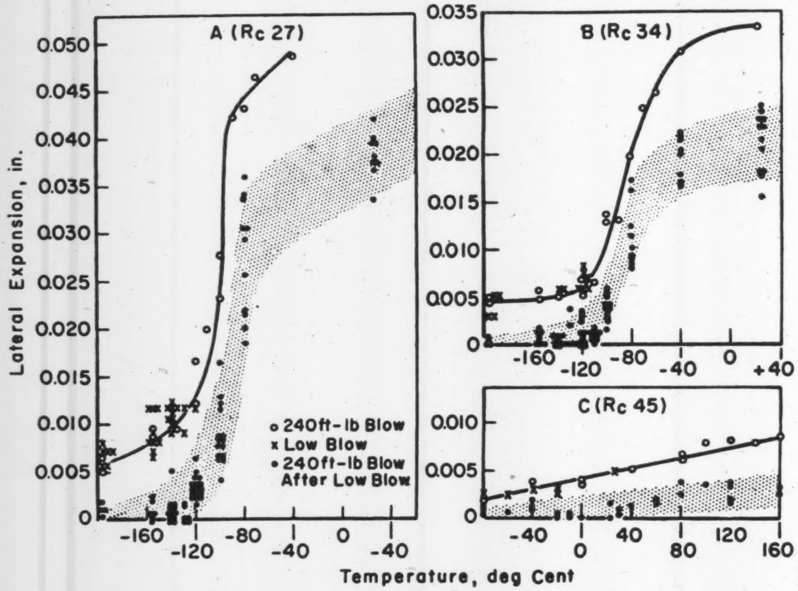


FIG. 13.—Deformation-Temperature Transition Curves for 4340 Steel at Three Hardness Levels

When the deformation (lateral expansion) as measured in a fractured test specimen (•) shows no increase over that measured after the initial blow, the data are plotted on the abscissa (zero deformation) and fracture is considered to be brittle. The data plotted as (*o*) and (*x*) represent total deformation in that the lateral expansion was measured in specimens which fractured under a single blow.

the pendulum, friction, etc. Such energy losses provide an explanation for the fact that the sum of the multiple-blow energies (low-blow energy plus the energy-to-fracture under a second blow) was generally greater than the energy absorbed in fracturing a test specimen with a single full-capacity blow (Fig. 12). Thus, only part of the energy in the impact machine pendulum is available for (1) doing plastic-deformation work and (2) developing elastic-stress energy in the test specimen in or near the path of fracture.

Lateral Expansion as a Measure of the Plastic-Deformation Work Required to Propagate the Initiating Crack:

By measuring the lateral expansion occurring in the test specimen after the initial blow and again after the fracturing blow, the difference provides a measure of the plastic-deformation work required to propagate the initiating crack. When the deformation as measured in the fractured test specimen shows no increase over that measured after the low blow, the plastic-deformation work to propagate the initiating crack is considered to be negligible. From Fig. 13, the highest temperature producing brittle fracture was −130 C for material condition A, −110 C for condition B, and 25 C for condition C. Separate plots for each of the different low-blow energies (20 and 30 ft-lb for condition A and 15, 20, and 25 ft-lb for condition B) and different deflection velocities (15 and 60-lb hammers to produce a given low-blow) indicated that variations in the magnitude and velocity of the initial low blow within the ranges investigated had no significant effect on the crack-propagation stage of fracture.

Multiple Low Low Blows to Simulate Cyclic Loading:

In a recent discussion of the brittle-fracture problem, Robertson (8) has pointed out that a ship " . . . which broke in two at dock . . . led to the realization that such fractures must not be attributed solely to high nominal stress values likely during storms and certainly not to general yield in the material prior to fracture, for the agreed nominal stress in the fractured plates was of the order of only 5 tons per sq in." The fact that several catastrophic service failures have occurred which appear to have involved little or no plastic deformation has been of prime interest to many investigators. Orowan (9), for example, has pointed out that in laboratory experiments with ductile steel, brittle fracture is initiated by large deformations producing strong plastic constraint and usually some fibrous cracking before cleavage cracking can start, whereas many service fractures seem to start without significant plastic deformation in spite of static loading.

Certainly at a nominal stress of 10,000 to 15,000 psi, general yielding would not be expected in ship steel. However, at points of stress concentration the yield strength may be exceeded at low nominal stress values. Moreover, strains caused by prior load cycles may have an influence on subsequent fracture. It is suggested by the author that due to the cyclic nature of most service, plastic deformation and macrocracking may occur in one cycle and then in a subsequent cycle involving a low value of nominal stress be followed by catastrophic brittle fracture. For example, a ship may be subjected to a storm at sea, return to port intact, and then having previously suffered plastic deformation at points of stress concentration, experience brittle fracture as a result of stresses developing from a sudden drop in temperature or shift in cargo.

An obvious question arising from this discussion concerns the effect of a succession of low blows (instead of a single low blow followed by a full-capacity blow as previously investigated). To determine the effect of multiple low blows, Charpy specimens were delivered initial blows both above and below the crack-initiating energy level (as determined by ink staining), followed by a succession of 10 ft-lb blows to fracture. Tests were conducted on material condition A at −100 and −140 C. The temperatures of −100 and −140 C were selected to bracket the brittle-fracture transition temperature. At −100 C, condition A withstood several low blows before fracturing, with each blow producing additional lateral expansion (Table I). At −140 C the material was not sufficiently ductile to withstand even one 10 ft-lb capacity blow subsequent to the initial blow; crack propagation occurred without measurable plastic-deformation work (Table II).

TABLE I.—MULTIPLE LOW BLOWS AT −100 C MATERIAL IN CONDITION A (ROCKWELL HARDNESS C 27)

Low Blow, ft-lb	Lateral Expansion, in.	
	Width	Increment
30	0.4080	0.0135
10	0.4130	0.0050
10[a]	0.4135	0.0005
30	0.4075	0.0130
10	0.4130	0.0055
10[b]	0.4145	0.0015
30	0.4090	0.0145
10	0.4110	0.0020
10	0.4160	0.0050
10[c]	0.4180	0.0020
20	0.4025	0.0080
10	0.4055	0.0030
10	0.4110	0.0055
10[d]	0.4095	0.0000
20	0.4020	0.0075
10	0.4040	0.0020
10	0.4070	0.0030
10	0.4110	0.0040
10[e]	0.4150	0.0040

[a] Fractured, 5.5 ft-lb absorbed.
[b] Fractured, 6.5 ft-lb absorbed.
[c] Fractured, 9.0 ft-lb absorbed.
[d] Fractured, 4.0 ft-lb absorbed.
[e] Fractured, 8.0 ft-lb absorbed.

If an analogy can be made between performance in the V-notch Charpy specimen and a point of stress concentration in a structure, the above results provide an explanation for service failures which have occurred at or below the design working stress. Explanation is based on the assumption that at some prior load cycle, sufficient strain occurred at a point of stress concentration to initiate micro-cracking. Subsequent cycles, even at loads well below the working stress, may then cause either progressive damage or sudden brittle crack propagation.

TABLE II.—MULTIPLE LOW BLOWS AT −140 C.

Low Blow, ft-lb	Lateral Expansion, in.	
	Width	Increment
20	0.4015	0.0070
10[a]	0.4010	0.0000
20	0.4020	0.0075
10[b]	0.4015	0.0000
20	0.4005	0.0060
10[c]	0.3995	0.0000

[a] Fractured, 3.5 ft-lb absorbed.
[b] Fractured, 4.0 ft-lb absorbed.
[c] Fractured, 4.0 ft-lb absorbed.

DISCUSSION OF RESULTS

Based upon a single heat of AISI 4340 steel, quenched and tempered to three hardness (strength) levels, the following behavior has been observed in V-notch Charpy specimens subjected to low-energy blows:

1. The initial cracking as produced by a low-energy blow occurred in the free surface of the machined V-notch on a shear stress trajectory.

2. While detection of this initial stage of cracking was difficult without metallographic examination, the second

stage of cracking was easily and reproducibly determined by ink staining. In the second stage, the initiating shear crack changed direction to propagate perpendicular to the principal stress and appeared in the fracture surface as typically fibrous.

3. The extent and ductile nature of the crack produced by a given magnitude of low blow was essentially constant over a wide range of temperatures extending below the brittle-fracture transition temperature.

4. The variation in deflection rate (approximately 4 to 16 ft per sec) introduced by the low-blow technique did not have an appreciable effect on the presence or extent of the initiating crack.

TABLE III.—TRANSITION TEMPERATURE.

Material Condition	Temperature, deg Cent		
	a	*b*	*c*
A (R_C 27)...........	−130	−120	−150
B (R_C 34)...........	−110	−120	−120
C (R_C 45)...........	25	25	120

[a] Brittle fracture under the second full-capacity blow.

[b] Brittle fracture under the initial low blow.

[c] Transition temperatures based on a 20 ft-lb energy level.

5. Specimens which were prestrained and cracked by low blows of different energy levels and then fractured over a range of testing temperatures by a full 240 ft-lb capacity blow, provided energy-to-fracture data which plotted as a single curve at temperatures producing crystallinity in the fracture surfaces. Thus, variations in the extent of the initiating crack, from microscopic shear to ink-stained fibrous cracks, had little or no effect on the amount of work necessary to propagate the initiating crack. Temperature was the controlling factor.

6. Using lateral expansion as a measure of plastic-deformation work, it was possible to distinguish between ductile and brittle fracture by measuring the lateral expansion associated with the crack-initiating low blow and then measuring the lateral expansion in the fractured test specimen. If, at the temperature of test, the initiating crack was propagated without measurable plastic-deformation work (no increase in lateral expansion after the initial blow), the fracture was classed as brittle. The highest temperature allowing brittle crack propagation was defined as the transition temperature. If, on the other hand, the crack-initiating low blow completely fractured the test specimen, this too was classed as brittle crack propagation; and the highest temperature permitting complete fracture under the initial low blow was also taken as the transition temperature. (The values as obtained by the two methods are listed in Table III.)

7. Investigation of the effect of multiple low blows offered an explanation for catastrophic service failures which have occurred under conditions of low nominal stress (10,000 to 15,000 psi). Specimens from material condition A (R_C 27) when subjected to a low blow of 20 ft-lb and subsequently struck a single 10 ft-lb capacity blow, fractured at −140 C with no measurable plastic deformation attending the second blow; whereas, when tested at −100 C, a number of 10 ft-lb blows were required to produce fracture, with plastic-deformation work attending each blow. Thus, an analogy is suggested between performance in the laboratory test specimen and the case of a bridge, ship, or storage tank subjected to a cyclic loading which causes local plastic yielding (at points of stress concentration) and then fractures in a brittle fashion on subsequent load involving low nominal stresses and temperature below the brittle-fracture transition.

CONCLUSION

An easily executed variation in the standard technique for testing V-notch Charpy impact specimens is presented. The technique permits a separation of the crack-initiation and crack-propagation stages of the fracture process. Differentiation between ductile and brittle fracture is based on the following concept: a brittle-fracture crack is propagated by continuous release of the elastic stress field surrounding the crack, whereas a ductile-fracture crack requires plastic deformation work to extend the crack. The measurement of lateral expansion (Poisson effect) provides a sensitive gage of the plastic deformation work done (1) in crack initiation and (2) in propagation of the initiating crack.

Two approaches appear feasible; both involve the initiation of a crack by means of an initial low-energy blow. One approach determines the highest temperature at which a crack-initiating low blow causes complete fracture of the test specimen, that is, the initiating crack propagates by continuous release of the elastic-stress field developed by the initial low blow. (In the case of the highly notch-tough material conditions A (R_C 27) and B (R_C 34), there is a critical low-blow energy level below which a self-propagating crack will not form.) The second approach involves the delivery of a crack-initiating low blow followed by a full-capacity fracturing blow, and is based on the concept that when the fracturing blow produces no measurable increase in lateral expansion, the plastic-deformation work required to extend the initiating crack is essentially zero; such a fracture is therefore considered brittle. The highest temperature permitting crack propagation without plastic-deformation work to extend the crack, is the brittle-fracture transition temperature. In that both approaches are based on the same criterion of brittle fracture, the transition temperatures determined by the two methods are, for all practical purposes, the same.

If, on the other hand, an arbitrary energy level, for example, 20 ft-lb, is used in determining transition temperature, the temperatures may be expected to be different from those determined on the basis of the brittle-fracture criterion (last column of Table III). It will be noted that the transition temperatures for material conditions A and C as determined by the 20 ft-lb energy level method do not check those obtained by the brittle-fracture criterion; whereas, the transition temperatures for condition B do check. It is believed that the reason for the latter is that the 20 ft-lb level is less arbitrary in the case of the condition B because 20 ft-lb is approximately the crack-initiating energy level for that condition. If the transition temperatures are determined for each material condition on the basis of the minimum energy to initiate cracking (that is condition A, 30 ft-lb; condition B, 20 ft-lb; and condition C, 13 ft-lb), the transition temperatures so obtained are approximately the same as those obtained by the brittle-fracture criterion.

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A TENSILE STUDY OF THE BRITTLE BEHAVIOR OF A RIMMED STRUCTURAL STEEL*

By E. T. Wessel[1]

Synopsis

The factors involved in the initiation of a brittle (cleavage) failure in a rimmed structural steel were investigated using notched and unnotched tension tests at temperatures down to −269 C. Cleavage fractures which occurred at all temperatures below 100 C were preceded by plastic deformation. The combined effect of increasing notch sharpness and increasing strain rate caused brittle failure to occur at higher temperature and lower stress levels. The notched tension results are correlated with Charpy "V" notched data. The results of the investigation are helpful in understanding the causes of brittle failure.

Unexpected brittle failure of mild steel structures is a serious problem to the nation as a whole. There are innumerable cases of brittle failures of a disastrous nature. Most publicized of these are the welded ship failures of World War II where over a thousand cases of severe damage as a result of brittle failures have been reported. Other steel structures have experienced the same phenomenon (**1**).[2] These include bridges, storage tanks, pressure vessels and pipe lines. Brittle failures are not confined to structural steels but also prevail in alloy steels as exemplified by recent failures of large generator and turbine rotors (**2**).

The susceptibility of metals to brittle failure cannot be ascertained from the conventional room temperature tension tests; however, several empirical laboratory tests have been devised to meet the needs of industry. Among these are the Charpy and Izod notched impact tests, the drop weight and explosion tests of Pellini (**3**), and assorted other notch tests. Basically these tests have been useful in evaluating the relative behavior of materials for specific applications but have been inadequate in providing an understanding of the mechanisms involved.

The low-temperature tension test was chosen as the key tool for this study into mechanisms of brittle failure because controlled brittle failures can be produced and quantitative measurements of the stresses and strains involved can be made. Primary emphasis is placed on the factors which govern the initiation

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 552.

of a brittle fracture rather than the propagation of the brittle crack once it has been initiated. The first phase of this program emphasizes temperature as the primary variable with major effort directed at obtaining quantitative measurements of the temperature dependence of the yield and brittle fracture strengths. Some preliminary studies of the combined effect of temperature, strain rate, and notch geometry are also included.

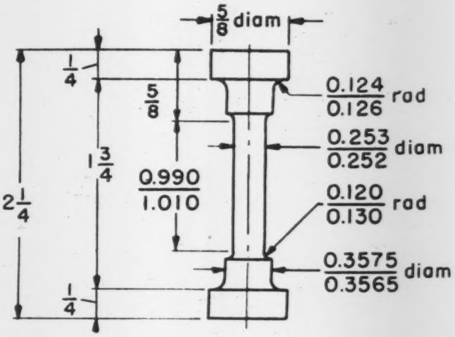
MATERIALS AND TEST PROCEDURE

Because of the widespread interest in ship failures and the apparent similarity of these failures to those occurring in other steel structures, the material chosen for this investigation was a rimmed steel plate kindly supplied by W. S. Pellini, Naval Research Laboratories. Some notched impact testing had already been done at the Naval Research Laboratories (4) using this particular heat of steel, (Code No. 31). The $\frac{3}{4}$-in. thick plate from which the samples were taken was rimmed steel in the as-rolled condition. This rimmed ABS-A steel is not representative of the currently used ship plate steel, but is, however, of the type previously used for ship construction where so many actual failures were experienced. For this reason the steel appeared to be particularly applicable for studies of the mechanism of brittle failure. The analysis of this steel was reported (4) to be as follows (weight per cent):

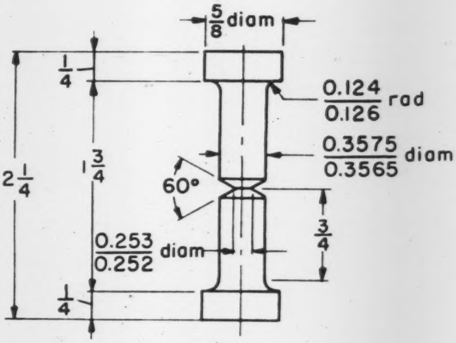
Carbon	0.24
Manganese	0.45
Silicon	0.02
Phosphorus	0.013
Sulfur	0.043
Nickel	0.12
Chromium	0.08
Copper	0.21

The microstructure of the plate was normal for this class of steel. It consisted of fine lamellar pearlite in a ferrite matrix. There was some ferrite banding and small voids and inclusions strung out in the rolling direction.

Tension specimens were taken with their axes parallel to the rolling direction of the plate. The specimen designs used in the study are illustrated in Fig. 1. The tension tests were conducted in a constant strain rate testing machine employing an autographic stress-strain recorder. The load is measured using a commercial SR-4 load cell (accuracy, ±0.25 per cent of capacity) and strain


5/8 diam
1/4
5/8
0.124/0.126 rad
0.253/0.252 diam
1 3/4
0.990/1.010
2 1/4
0.120/0.130 rad
0.3575/0.3565 diam
1/4
Approximate Elastic Stress Concentration Factor, Kt, at Gage Section Fillet = 1.18

Unnotched


5/8 diam
1/4
0.124/0.126 rad
0.3575/0.3565 diam
2 1/4
1 3/4
60°
3/4
0.253/0.252 diam
1/4
Group No. 1 - Rad 0.010", Kt = 3.3
Group No. 2 - Rad 0.0005", Kt = 13.2

Notched

FIG. 1.—Test Specimens.

is measured by a direct mechanical linkage of the recorder chart drive to the test machine drive (crosshead movement). The arrangement is such that plastic strains as small as 0.01 per cent (1×10^{-4} in. per in.) can be determined from the autographic stress-strain curves. The sub-zero test temperatures are achieved and maintained utilizing apparatus and techniques which have been previously described (5, 6). Conventional procedures were used for the moderately elevated temperature tests.

Care was exercised to obtain good axial alignment of the test specimen to minimize scatter induced by bending moments resulting from non-axial loading. Some of the features incorporated were: The use of special universal joints in the specimen holder system, precision machining of specimens and all the related gripping components, and the use of shoulder loaded type test specimens to afford some further opportunity for self alignment.

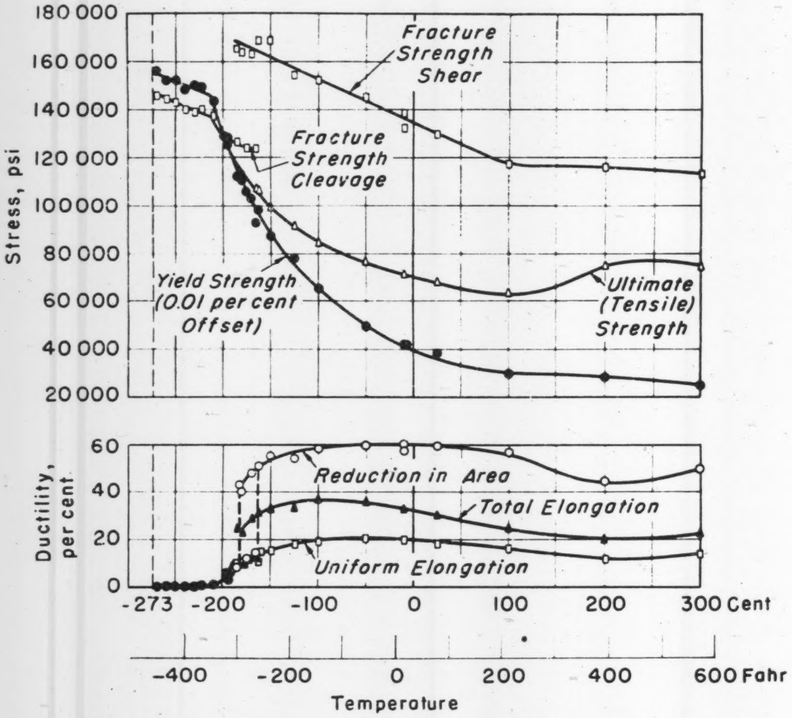


FIG. 2.—The Effect of Temperature on the Conventional Tension Properties of a Rimmed Structural Steel.

TEST TESULTS

Unnotched tension specimens were tested at a constant-strain rate of 2.1×10^{-3} per sec over the temperature range from 300 C (572 F) to −269 C (−452 F). The results of these tests are shown in Fig. 2 where the conventional strength and ductility criteria are plotted as a function of test temperature. Of major interest is the pronounced transition from a ductile-to-brittle behavior which occurs in the temperature range between −165 C (−265 F) and −185 C (−283 F). This transition is characterized by a sharp decrease in ductility, a pronounced decrease in fracture strength, $\left(\frac{\text{load}}{\text{final area}}\right)$, a change in the mode of fracture initiation from shear to cleavage, and the absence of any general localized deformation (necking). In this transition zone there is an extremely fine balance between fracture initiation by shear or by cleavage; either type can occur with corresponding large differences in ductility and fracture strength.

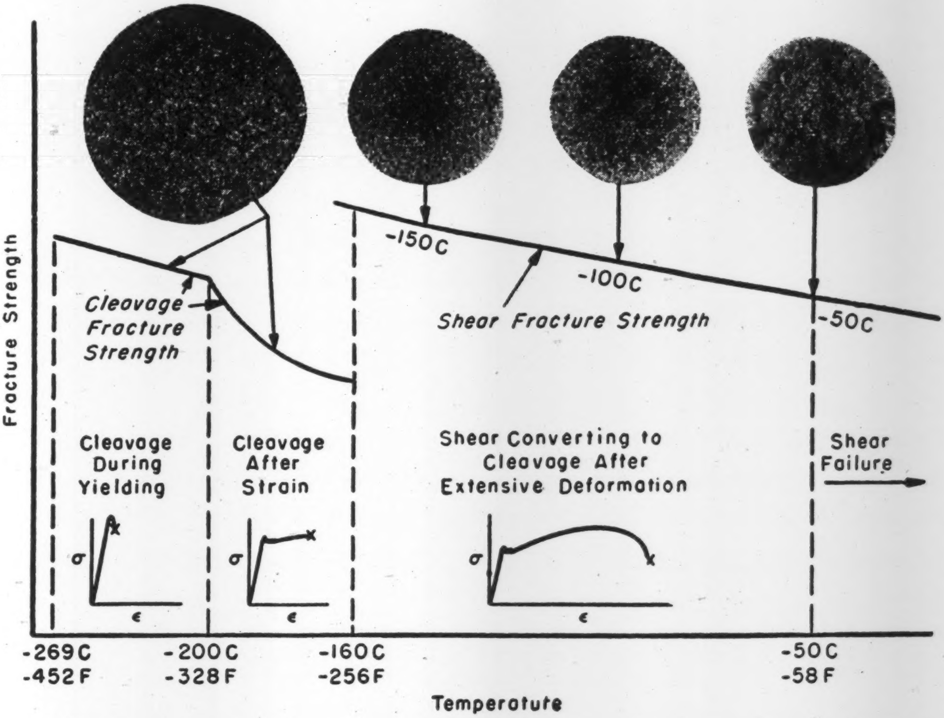

Fracture Strength
-150C
-100C
-50C
Shear Fracture Strength
Cleavage Fracture Strength
Cleavage During Yielding
Cleavage After Strain
Shear Converting to Cleavage After Extensive Deformation
Shear Failure
σ
ε
x
-269C -452F
-200C -328F
-160C -256F
-50C -58F
Temperatute

FIG. 3.—Comparison of Fracture Appearance and Type of Stress-Strain Curves for Various Test Temperatures.

In the temperature range below −200 C (−328 F), the specimens yield and fracture occurs during this abrupt yielding; hence the yield strength is greater than the fracture strength. This behavior is somewhat contrary to the general concept of brittle fracture. It is believed that below −200 C the initiation of sharp yielding is caused primarily by microcracking rather than slip and that fracture occurs when these microcracks reach sufficient size to become self-propagating.[3] The yield strength exhibits the usual strong temperature dependence characteristic of body-centered-cubic metals. However, it was observed that the yield strength does not continue to rise to extremely high values at the lower temperatures but exhibits a definite change to a much weaker temperature dependence below −200 C. This behavior of yield strength has been observed in many materials of various lattice structures and will be described and discussed in detail elsewhere (7).

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[3] Paper on "Mechanism of the Yield Point and Brittle Fracture" is being prepared to discuss this aspect in more detail.

For the purpose of discussing brittle failure, the curve of fracture strength as a function of temperature holds most interest. The fracture strength is replotted as a function of temperature in Fig. 3 together with illustrations of typical stress-strain curves and photographs of the fractures. There are three distinct types of brittle failure. At the lowest temperatures, from −269 C (−452 F) to −200 C (−328 F), brittle cleavage fracture occurs during the initial abrupt yielding and is preceded by only very small amounts of plastic strain. Since these small strains are relatively constant, the slope of the fracture strength curve is primarily an indication of the temperature dependence of cleavage fracture strength. Eldin and Collins (8) observed a similar temperature dependence of cleavage fracture strength in the same temperature range.

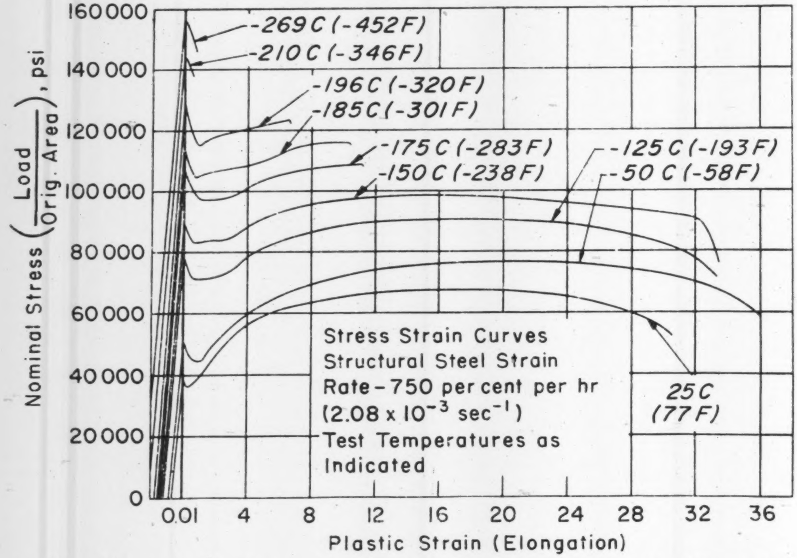

FIG. 4.—The Effect of Test Temperature on the Stress-Strain Curves of a Rimmed Structural Steel.

A second type of brittle failure occurs in the temperature range from −200 C (−328 F) to −160 C (−256 F). In this temperature range the steel is capable of considerable plastic deformation (as much as 14 per cent elongation) which terminates in a completely cleavage-type of fracture. The slope of the fracture strength curve in this temperature range is a result of combined temperature and strain effects. In this temperature range, cleavage failure occurs when microcracks, which are formed either during initial yielding or during subsequent plastic flow, either grow in size or several join together to form a critical size sufficient to become self-propagating. Plastic strain, in this temperature range at least, causes permanent fracture damage to the steel.

The third type of brittle failure occurs in the temperature range from −160 C

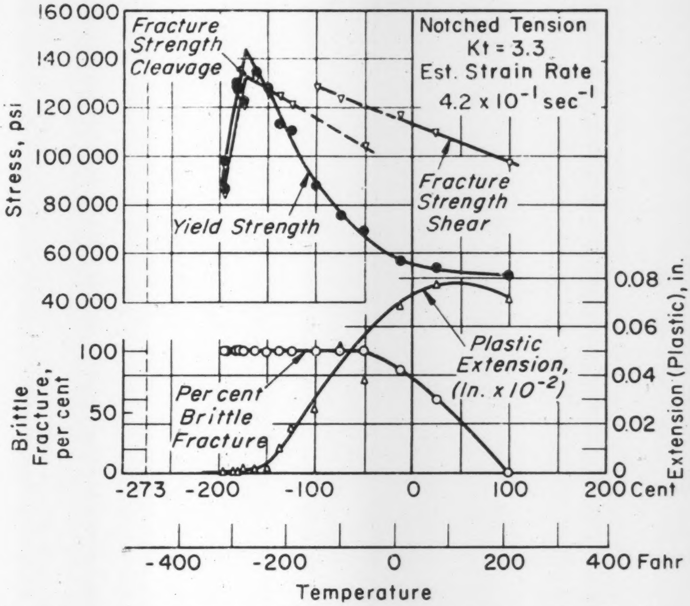

FIG. 5.—The Effect of Temperature on the Notched Tension Properties of a Rimmed Structural Steel. Modest Notch and Strain Rate.

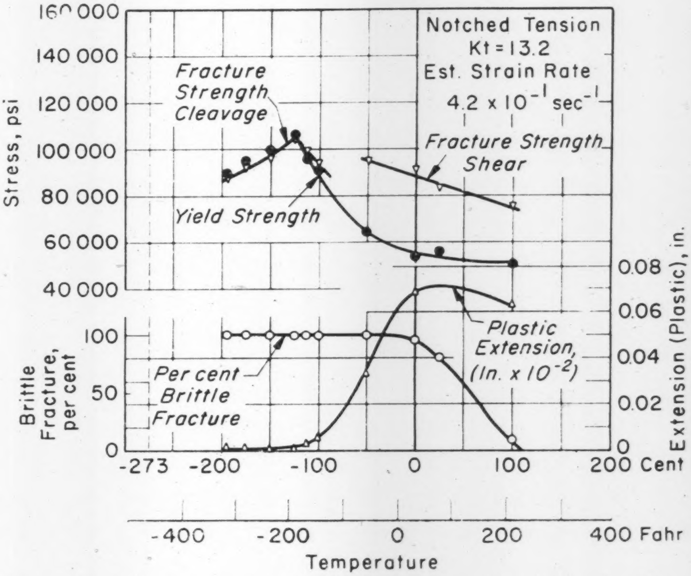

FIG. 6.—The Effect of Temperature on the Notched Tension Properties of a Rimmed Structural Steel. Severe Notch, Modest Strain Rate.

(−256 F) to −50 C (−58 F). In this range the steel is capable of undergoing much larger amounts of deformation including necking. Failure initiates with a fibrous shear fracture in the center of the specimen and then converts to cleavage fracture. The amount of shear in the total fracture area increases with increasing temperature, being a small fibrous dot in the center of the specimen at −160 C and increasing to all shear above −50 C. The shear fracture strength curve in this temperature range is believed to represent the stress required to initiate a shear failure.

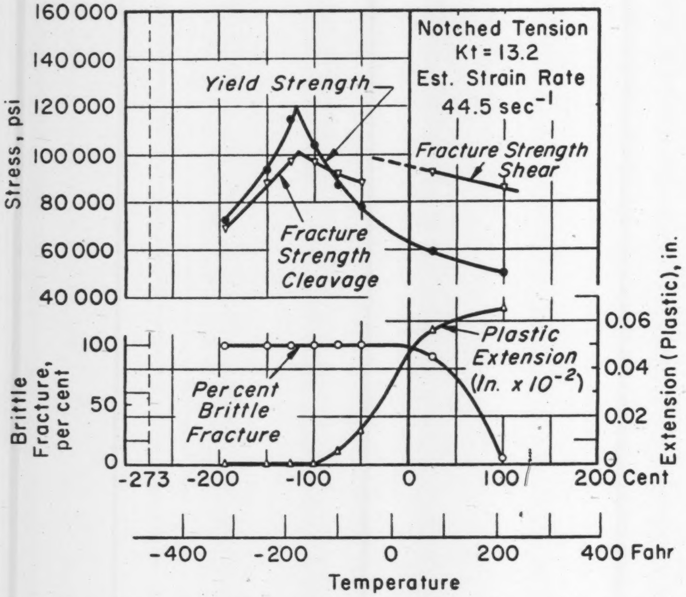

FIG. 7.—The Effect of Temperature on the Notched Tension Properties of a Rimmed Structural Steel. Severe Notch, Fast Strain Rate.

Actual stress-strain curves for various temperatures are shown in Fig. 4. The curves for −50 C, −125 C, and −150 C are typical of those where fracture initiates as shear and converts to cleavage. They are not unlike that for total shear failure (25 C). The curves for −175 C, −185 C, and −196 C illustrate the stress-strain curves where considerable plastic deformation precedes a total cleavage failure. Stress-strain curves associated with the third type of brittle failure are illustrated in curves (−210 C and −269 C) where complete cleavage failure occurs during the initial abrupt yielding.

Having established the influence of temperature on the ductile-to-brittle behavior for uniaxial tension, the effects of triaxial stresses (notches) and high strain rates were investigated since these factors are known to raise the transition temperature. Three sets of notched experiments, as outlined below, were conducted over a range of sub-zero temperatures:

Group	Notch Radius, in.	Elastic Stress Concentration, Factor, K_t	Estimated Strain Rate, per sec
A.......	Unnotched	1	2.1×10^{-3}
B.......	0.010	3.3	4.2×10^{-1}
C.......	0.0005	13.2	4.2×10^{-1}
D.......	0.0005	13.2	44.5

The strain rates indicated are based on an estimation of the effective gage length at the root of the notches and the actual head rate of the testing machine. The assumed gage length of $\frac{1}{2}$ of the notch radius provides a reasonable approximation only for the early stages of plastic flow.

The results of the series of notched tension tests are shown in Figs. 5 to 7. The yield strength shown in these curves is defined as the flow stress at 1×10^{-4} in. of plastic extension. As was the case for unnotched tension tests, three types of brittle failure were observed. At the lowest temperatures, cleavage failure occurred during an initial discontinuous yielding. At intermediate temperatures, considerable plastic deformation preceded cleavage fracture. At still higher temperatures, the failures initiated as a shear fracture at the *root* of the notch and converted to cleavage, the failure being preceded by rather extensive localized plastic deformation. Felbeck and Orowan (9) have observed this latter type of brittle failure in notched tension tests at room temperature and attribute the change from shear to cleavage to a high velocity dependence of the yield stress acting at the tip of the propagating shear crack. The present study indicates that the strain dependence of the cleavage fracture strength may also be a major factor in promoting cleavage failure. Thus the conversion from shear to cleavage fracture is probably the result of the accumulative effects of increasing strain rate and decreasing cleavage fracture strength in the region undergoing plastic deformation at the advancing tip of the crack. The first evidence of the transition from a cleavage to a shear fracture initiation behavior is difficult to distinguish by the appearance of the notch fractures because of the large amount of localized deformation and the narrowness of the shear lip at the root of the notch.

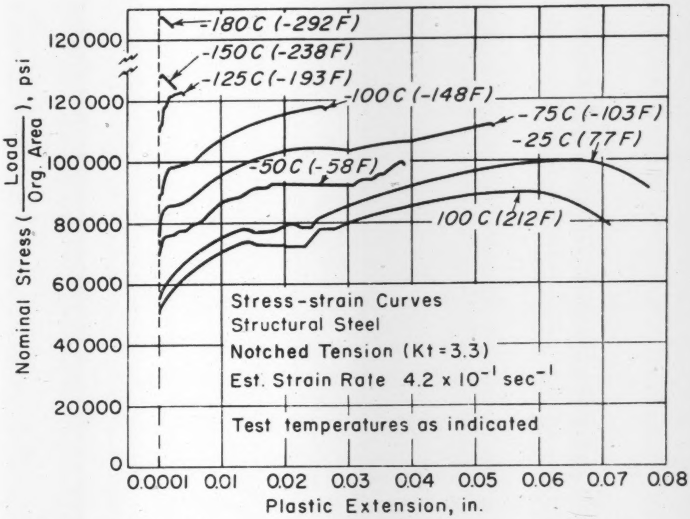

FIG. 8.—The Effect of Test Temperature on the Stress-Strain Curves of a Rimmed Structural Steel Tested in Tension in the Notched Condition.

With one exception, the notched and unnotched data follow the same pattern. This exception is in the slope of the yield and fracture strength curves in the temperature range below their intersection (−200 C—unnotched, −150, −125, −75 C—notched). The unnotched data indicate a decreasing yield and fracture strength with increased temperature and the notched data show the opposite effect. The reason for this has not conclusively been determined but is probably due to the fact that at temperatures below these intersection points the stress and strain concentrations are not readily relaxed and the steel behaves more and more as predicted by the elastic theory of notches.

Some typical stress-strain curves for the notched tension tests are shown in Fig. 8. The irregularities in the curves apparently are due to inhomogeneous flow resulting from changes in plastic constraint due to changes in the shape of the notch following relatively large plastic deformations. The curves illustrate a relatively rapid rise of flow stress with increasing plastic strain. Some tendency for discontinuous yielding is also observed.

The effect of temperature, strain rate, and notch geometry on the flow and fracture characteristics are summarized in Fig. 9. In general it is shown that increasing the severity of test conditions, that is, sharper notches or higher strain rates, will cause brittle failure to occur at higher temperatures and lower stresses.

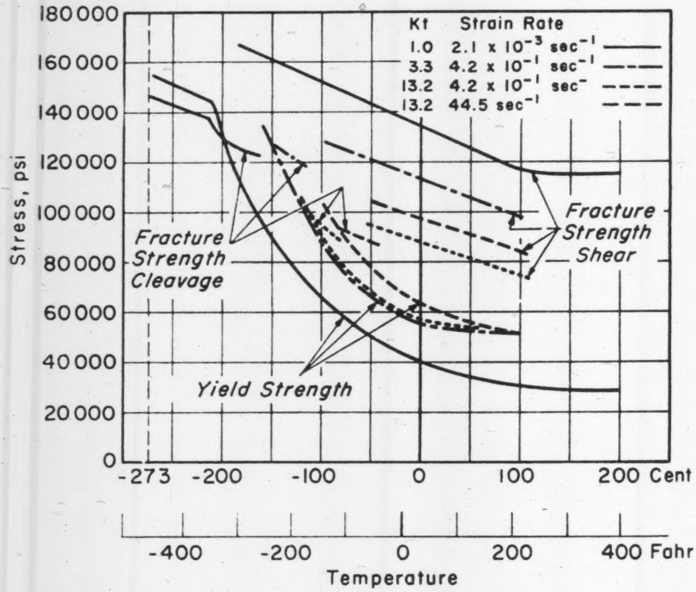

FIG. 9.—The Effect of Temperature, Strain Rate, and Notch Geometry on the Flow and Fracture Strengths of a Rimmed Structural Steel.

The individual roles of the various factors can be discussed in more detail.

The yield strength appears to be influenced very little, if any, by changes in the sharpness of the notch. This is illustrated by comparing the yield strength curves (Fig. 9) of group B with group C which had different notch severities, $Kt = 3.3$ and $Kt = 13.2$ respectively but equivalent strain rates of 4.2×10^{-1} per sec. Strain rate appears to be the predominant factor in producing a shift of the yield strength curve. This can be seen by comparing the yield strength curves (Fig. 9) of group C with group D which had equivalent notches, $Kt = 13.2$, but different strain rates, 4.2×10^{-1} per sec and 44.5 per sec respectively.

The effects of strain rate and notch sharpness on the cleavage fracture strength are exactly the opposite from their relative effects on yield strength. Strain rate has relatively little effect whereas notch sharpness is very important. The small effect of strain rate can be seen by comparing the cleavage fracture strengths (Fig. 9) of groups C and D which had equivalent notch sharpness, $Kt = 13.2$, but different strain rates of 4.2×10^{-1} per sec and 44.5 per sec, respectively. The pronounced effect of notch sharpness on fracture strength can be seen by comparing curves for groups B and C which had equivalent strain rates, 4.2×10^{-1} per sec, but different notch severities, $Kt = 3.3$ and $Kt = 13.2$, respectively. The general indications are that mild notches, $Kt = 3.3$, do not cause much change in the cleavage fracture strength level, whereas more severe notches, $Kt = 13.2$, have a considerable effect. The resulting effect of the two variables—strain rate increasing yield strength and notch severity lowering fracture strength—working together causes brittle failure to occur at higher temperatures and lower stress levels.

The effects of notch geometry and strain rate on the shear fracture strength (initially shear converting to cleavage) curves are quite pronounced. Increased sharpness of the notch lowers the shear fracture strength while increased strain rate for a given notch severity increases the shear fracture strength.

Because of the widespread interest in the use and interpretation of Charpy V-notch impact data, a comparison of Charpy impact and notched-tension data is shown in Fig. 10. Within the limits of experimental error in determining the relative amount of shear and cleavage components in a fracture surface, the curve of per cent cleavage fracture as a

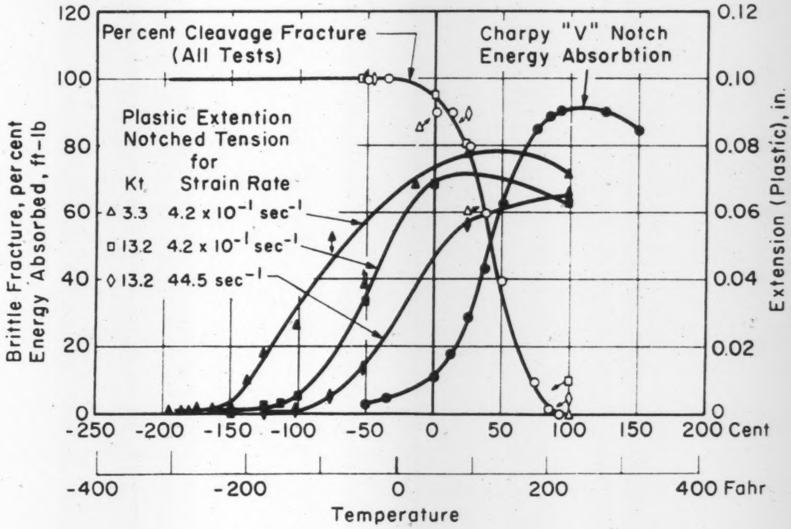

FIG. 10.—Comparison of Charpy V-Notch Impact Data with Notched Tension Results for a Rimmed Structural Steel.

function of temperature is essentially the same for the Charpy and all of the notched-tension tests. In the light of this observation, if one were to use a fracture appearance criterion for the determination of the transition temperature, the notched-tension test would, in the author's opinion, be a better test than the Charpy V notch impact test since the tension data would also provide quantitative measurements of the stresses and strains as well as the type of brittle fracture at various points on the transition curve. In considering the plastic extension *versus* temperature curves of Fig. 10, it appears that with sufficiently high strain rates or severe notches a more positive correlation of notched tension and Charpy impact data can be obtained. The energy absorption (area under stress-strain curves) for the notch-tension data when plotted against temperature provides essentially the same curves as the plastic extension criterion used in Fig. 10.

DISCUSSION

From the practical viewpoint it is necessary to correlate the results of these experiments on brittle fracture initiation with recent work concerning the propagation of brittle fracture. The data previously described show that increasing the severity of the notch or increasing the strain rate causes all types of brittle fracture to occur at higher temperature levels and at considerably lower stress levels. An approximate indication of the general effect of this trend is shown in Fig. 11 where the stress required to initiate cleavage fracture after some prior macroscopic deformation is shown as a function of temperature. If it is assumed that further increases in notch sharpness or strain rate would continue this trend to higher temperatures, an extrapolation of the curve indicates that in the presence of very severe conditions cleavage fracture could be initiated at room temperature by average stresses of approximately 50,000 psi. Once the failure is

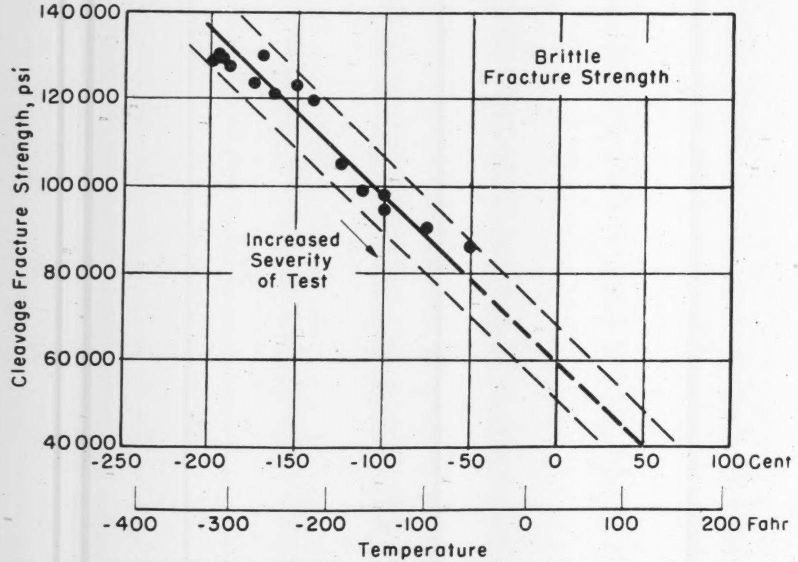

FIG. 11.—Approximate Relationship Illustrating the Effect of Increasing Severity of Test Conditions on Brittle Fracture Strength.

initiated it can readily propagate in a brittle manner throughout the structure at much lower stresses than are required for its initiation. Feely, *et al.* (**10, 11**) have quite capably demonstrated that a brittle cleavage fracture, once initiated, can be readily propagated by relatively low stresses. A steel of the same general type and composition as that used in the present investigation was found (**11**) to have a "cleavage fracture propagation strength"[4] of 18,000 psi at 50 F. For any given steel, cleavage propagation stress decreases with decreasing temperatures.

In the light of the foregoing discussion, one can readily conceive how brittle failures of mild steel structures can and do occur in practice. First it is quite reasonable to assume that extremely sharp notches, for example, welding cracks, inclusions, voids, etc., are present where localized strain could occur at very high strain rates. These points provide ready nuclei for the initiation of a brittle failure. The critical conditions necessary to initiate failure at these trigger points are severe but not unreasonable. The data indicate that, for certain classes of mild steel, brittle failure at room temperature can be initiated by average stresses of approximately 50,000 psi when the necessary severity of conditions exists. These critical conditions of stress, strain, strain rate, and notch severity for fracture initiation are quite dependent upon temperature. Therefore, slight decreases in temperature or slight increases of stresses and localized strain or a combination of the two would be sufficient to trigger these existing nuclei. Once initiated, the stresses provided by normal static loading conditions are sufficient to readily propagate the brittle failure. These propagation stresses have been found (**10, 11**) to be quite low (18,000 psi and less) in certain classes of steels for the temperature range (at or below room temperature) encountered in practice.

SUMMARY AND CONCLUSIONS

The tensile properties of a rimmed structural steel have been determined over a wide range of temperatures extending from −269 C (−452 F) to above room temperature. Three distinct types of brittle (cleavage) failures were found to occur in both notched and unnotched tests. These are characterized by the following: (1) at low temperatures, cleavage failure occurred during initial discontinuous yielding; (2) at intermediate temperatures, cleavage failure occurred after moderate amounts of plastic strain; (3) at higher temperatures, the material could withstand a considerable amount of deformation, fracture then initiating as shear and converting to cleavage. At still higher temperatures, complete shear failures were encountered.

The combined effect of increased notch sharpness and increased strain rate caused all three types of brittle failures to occur at considerably higher temperatures and considerably lower stress levels. The geometry (sharpness) of the notch alone had no effect on the initial yield strength in the temperature range where some macroscopic deformation could occur. Fracture strength decreased markedly with increasing sharpness of the notch, probably as a result of the strain-induced damage in the highly localized region undergoing deformation. Increased strain rates for a given notch sharpness produced an appreciable rise in yield strength and relatively little effect on fracture strength. The results of the study indicate that, for the temperature range encountered in practice, average stresses of the order of 50,000 psi would be sufficient to initiate total cleavage fracture in the presence of extremely sharp notches where the strain would be highly localized and the associated strain rates would be relatively fast.

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[4] Terminology which the author has applied to the results of Feely, *et al.* (**10, 11**).

Acknowledgments:

The author is indebted to his associates at Westinghouse for many helpful and stimulating discussions. He is especially indebted to W. H. Pryle, Metallurgy Dept., Westinghouse Research Laboratories, for conducting the experimental tests and to J. H. Bechtold, the Manager of the Department, for his interest in this work. Further appreciation is expressed to W. S. Pellini, Superintendent, Metallurgy Division, Naval Research Laboratory, for supplying the necessary ship plate steel, and to the Cryogenics Section of Westinghouse Research Laboratories for supplying the necessary liquid helium.

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DISCUSSION

Mr. George Sachs.[1]—The author illustrates an interesting relation between the transition temperatures derived from impact tests and static notch-tension tests, respectively. As the strain rate decreases, the transition temperature changes to lower values. There also appears to exist an effect of stress concentration or notch sharpness which is difficult to understand. Armstrong and Gagnebin[2] have already shown, some time ago, that the transition temperature for impact tests is reduced, as the notch radius is increased or the stress concentration is decreased.

Mr. E. T. Wessel (*author*).—As Mr. Sachs points out, a decrease in the effective strain rate in the notch tension test results in a lowering of the transition temperature as indicated by the total plastic extension criteria, Fig. 10. As was described in more detail in the paper, the lowering of the transition temperature by decreasing the strain rate is largely the result of the effect of strain rate on yield strength—a decrease in strain rate effectively causing a displacement of the yield strength *versus* temperature curve to lower temperature levels.

The basic means by which an increase in the notch radius (decrease in notch sharpness or theoretical stress concentration) causes a lowering of the transition temperature is not as clearly seen. Normally this behavior is attributed to the effects of the corresponding changes in stress concentration and their subsequent influence on the measured or average applied stress level which is required to initiate yielding or fracture. However, as illustrated in Fig. 9, when specimens of varying notch radii (notch sharpness or stress concentration) are tested at the same effective strain rate, no difference in yield strength (defined as the stress at the minimum observable plastic extension of 1×10^{-4} in.) can be ascertained. Hence the primary means by which notch sharpness affects the transition temperature is by its relative influence on the measured or average fracture strength—decreasing notch sharpness increases the fracture strength, lowering the transition temperature, for tests at the same effective strain rate. This behavior is attributed to a smaller degree of strain concentration or plastic restraint in the less sharp notches.

Stress concentrations, as are normally considered to exist at the root of a notch, may influence the applied stress level required for the very early stages of plastic deformation; that is at plastic strains smaller than the minimum observable amount of 1×10^{-4} in. detectable with the apparatus used in these experiments. Even if such effects do prevail in the microstrain region, they apparently exert little influence on the general flow behavior for the temperature range in which the steel is capable of relatively larger amounts of notch ductility before brittle fracture. However, at temperatures below the intersection of the yield and fracture curves (Figs. 5 to 7) where the notch ductility is quite small and brittle fracture occurs during initial yielding, notch stress concentrations appear to have a pronounced effect on the measured average yield and fracture stresses, and the data indicate a closer approach to the results anticipated from elastic theory.

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[1] Associate Director of Research, Syracuse University Research Inst., Syracuse, N. Y.

[2] T. N. Armstrong and A. P. Gagnebin, "Impact Properties of Some Low-Alloy Nickel Steels at Temperatures Down to −200 F," *Transactions*, Am. Soc. Metals, Vol. 28, pp. 1–19 (1940).

The author is indebted to Mr. Sachs for having pointed out the difficulty in understanding the effects of notch sharpness on the transition temperature and for the reference to the work of Armstrong and Gagnebin[2] which correlates quite well with the findings of the present investigation.

EFFECTS OF A NUMBER OF HEAT TREATING AND TESTING VARIABLES ON THE NOTCH STRENGTH OF 4340 STEEL*

BY G. SACHS,[1] V. WEISS,[1] AND E. P. KLIER[1]

SYNOPSIS

Tests on sharply notched bars performed at sufficiently low temperature clearly disclose the tendency of 4340 steel to be highly notch sensitive on static loading, if tempered at temperatures between 500 and 700 F. These tests also reveal that a two-step quenching procedure leads to high-strength steel conditions that are superior to those obtained on conventional oil quenching and tempering.

In the evaluation of the notch sensitivity of high-strength steels, the notch-tension test is now widely used as an index of service behavior. For a given steel, however, many factors affect the notch-strength measured in the test. Some of the most important of these are the following:

(*a*) The metallurgical structure, resulting from tempering the martensite, bainite, or mixed structures in the "500 F embrittlement" range,[2]

(*b*) The specimen size and notch geometry, and

(*c*) The testing temperature and the strain rate.

It is now generally accepted that "slack-quenched" structures give rise to inferior notch properties compared to initially martensitic structures when tempered to comparable strength levels (**1**).[3] It has become an increasingly important objective, therefore, to avoid slack-quenching of steels for high-strength use. Heretofore, this has been possible only by the use of ultra-deep-hardening steels.

While slack-quenched steels have deficient notch properties, the principal low-alloy steel for aircraft applications, namely, 4340 steel, if transformed to lower bainite, has properties comparable to and perhaps even superior to the properties of tempered martensite at the same strength level (**2**). Such bainitic structures, however, will not attain the highest strength levels obtainable for tempered martensite which are desired. Recently, it has been shown that 4340 steel by suitable thermal cycling can be transformed on quenching to high-strength bainite (**3**). The notch-strength properties of such high-strength bainite are at present unreported.

The objective of the investigation to be discussed was threefold. First, the

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

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[2] Actually, minimum ductility and impact strength are usually observed on tempering between 600 and 700 F.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 565.

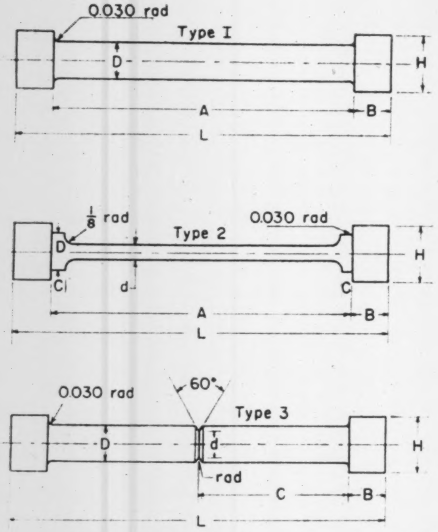

FIG. 1.—Test Specimens. For Dimensions See Table I.

TABLE I.—MAJOR SPECIMEN DIMENSIONS, NOTCH RADII AND STRESS CONCENTRATION VALUES. (SPECIMEN ILLUSTRATED FIG. 1.)

DIMENSIONS, IN.

	Unnotched			Notched
	Type 1	Type 2	Type 3	Type 4
A	1.00	1.00	4.00	...
B	0.25	0.25	0.50	0.25
C	...	0.150	...	0.50
D	0.300	0.300	0.500	0.300
d	...	0.200	...	0.212
H	0.50	0.50	0.75	0.50
L	1.50	1.50	5.00	1.50

STRESS CONCENTRATIONS K_t, SEE PETERSON PAPER (6).

r	0.010 in.	0.005 in.	0.001 in.	0.0005 in.	0.0004 in.	0.0003 in.	0.0002 in.	0.0001 in.
$D = 0.3$	3.2	4.2	8.8	12	13.5	15.5	18.5	26
$D = 0.5$	3.9	5.2	11	15.5	...	...	...	...

general response of various strength levels of 4340 steel to stress concentrations of different magnitude and at testing temperatures ranging from room temperature to very low temperature was to be evaluated. Second, from such tension and notch-tension tests, the range of existence of the 500 F embrittlement under static loading conditions was to be determined. Third, a determination was to be made of the effects of two-step treatments promoting lower bainite structures on the notch-strength characteristics and 500 F embrittlement of 4340 steel.

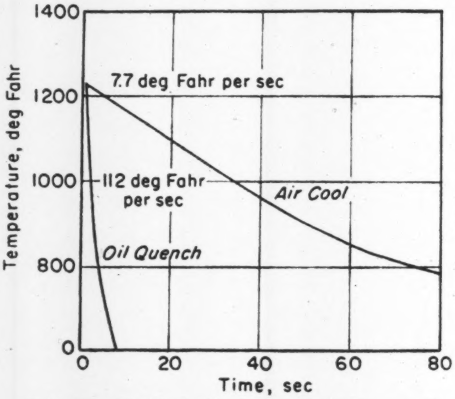

FIG. 2.—Typical Cooling Rate Curves for Notch Tension Specimens, 0.3 in. Diameter.

EXPERIMENTAL PROCEDURE

Material:

The 4340 steel studied was supplied as $\frac{7}{8}$-in. bar stock of the following composition:

Carbon, per cent	0.38–0.39
Manganese, per cent	0.76
Phosphorus, per cent	0.015
Sulfur, per cent	0.015
Silicon, per cent	0.29
Nickel, per cent	1.84
Chromium, per cent	0.72
Molybdenum, per cent	0.23

Heat Treatments:

The 4340 steel was heat treated in three different manners, as follows:

The conventional heat treatment was to normalize from 1600 F, austenitize at 1600 F for 1 hr, oil quench and temper

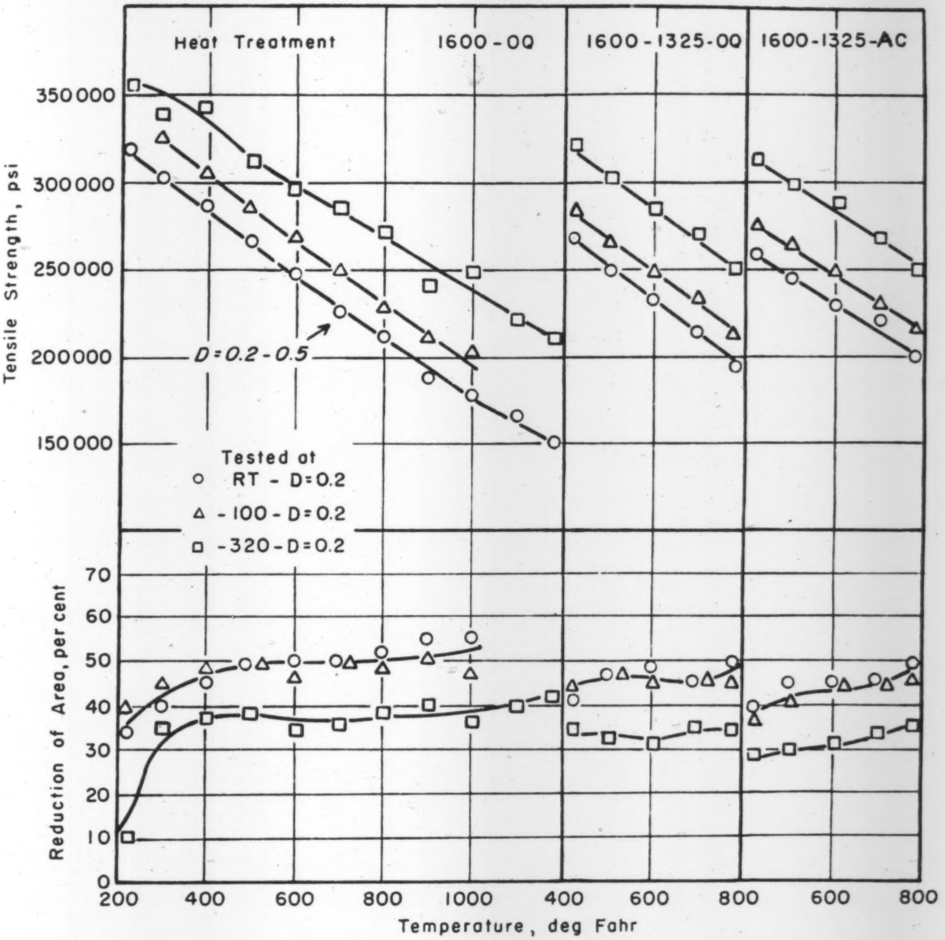

FIG. 3.—Tensile Strength Levels of 4340 Steel as a Function of Heat Treatment and Testing Temperature.

for 1 hr. The tempering temperatures usually ranged from 400 to 800 F and were occasionally as low as 212 F and as high as 1200 F.

The two additional heat treatments were aimed at forming lower bainite on cooling. These specimens were heated to 1600 F for 1 hr, furnace cooled to 1325 F and held at this temperature for 1 hr. One set of specimens was then oil quenched; the second was air cooled.

Typical cooling-rate curves for the center of notched sections were recorded for the last two heat treatments and are given in Fig. 2. These rates at 1250 F correspond to Jominy distances of about $\frac{3}{16}$ and $1\frac{3}{16}$ in., respectively. Therefore, the transformation products resulting from both cooling cycles should be approximately the same.

Test Specimens:

Both smooth and notched tension-test bars, shown in Fig. 1, were tested. Variations in major dimensions are listed in Table I.

Test Conditions:

The specimens were loaded in special adaptors to insure concentricity. Tests were performed at room temperature (approximately 80 F), in dry ice and acetone (approximately −100 F), and in liquid nitrogen (approximately −320 F).

Representation of Test Data:

Average points, as a rule from two tests, are reported in graphs. Due to the comparatively small number of tests for each particular condition, some scatter has remained in the average results, particularly for tests at −320 F. The construction of trend curves through these points has been made to insure that all curves are in conformance to each other and to the general rules established by the large total volume of the work.

EXPERIMENTAL RESULTS

Effect of Testing Temperature on Tensile Properties:

The results of tension tests on 4340 steel at three different temperatures after the various heat treatments are presented in Fig. 3. In all instances, a uniform decrease in the testing temperature resulted first in a small and then in a comparatively large increase in tensile strength.

The ductility was practically the same at −100 F as at room temperature, but comparatively lower at −320 F. This difference was 20 to 25 per cent for all heat treatments and tempering temperatures of 300 F or higher. However, for the oil-quenched condition after the 212 F temper, the ductility at −320 F was greatly reduced, giving rise to a ductility transition at a tempering temperature between 200 and 300 F.

Therefore, only a slight indication of the 500 F embrittlement, represented by a small decrease in ductility at tempering temperatures between 500 and 700 F, was encountered in the regular tension tests on the 4340 steel at any of the temperatures investigated. However, accurate tests on high hardenability steels usually reveal a slight depression in the reduction of area values of unnotched specimens after tempering at approximately 700 F (5). This has not yet been explained and may be an indication of the development of the 500 F embrittlement.

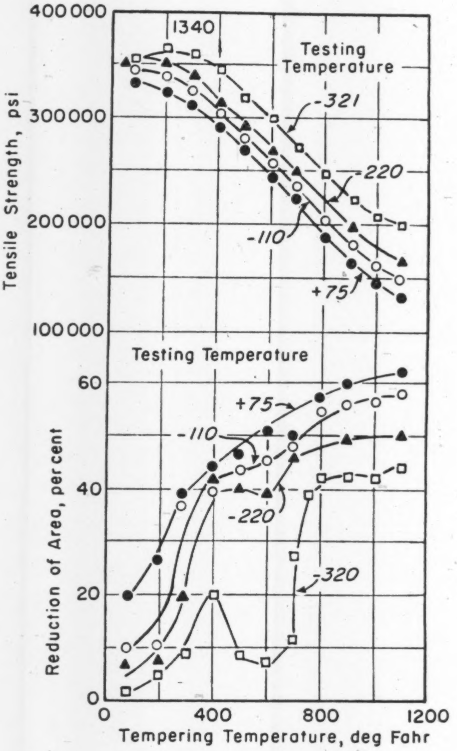

FIG. 4.—Tensile Strength Levels of 1340 Steel as a Function of Testing Temperature.

This is in distinct contrast to the previously observed behavior of a steel possessing lower hardenability, namely, a 1340 steel, which, because of its inferior low temperature properties, suffers a great loss in ductility on testing below −220 F (see Fig. 4) if tempered at temperatures between 500 and 700 F (4).

The tensile strength of heat-treated steels has been found in earlier tests on materials at strength levels up to about 180,000 psi to increase with decreasing temperature at a rate that depended primarily upon the tensile strength and became smaller as the strength increased (4), as shown in Fig. 5. This graph also includes the results of present tests and shows that the relative increase in strength on lowering the testing temperature, is further reduced as the tensile strength increases up to nearly 300,000 psi. This is further illustrated in Fig. 6, as a function of the tempering temperature.

Basic values of tensile strength were needed for the evaluation of stress-concentration effects. In order to eliminate the unavoidable variations in the experimental data, such basic values were obtained from the various trend curves in Figs. 3 and 6.

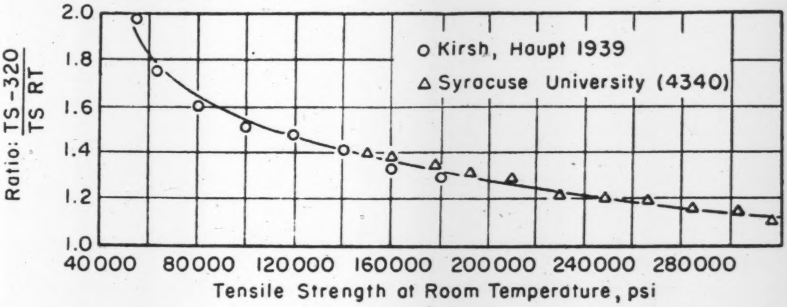

FIG. 5.—Effect of Strength Level on the Low Temperature Strength of High Strength Low Alloy Steels.

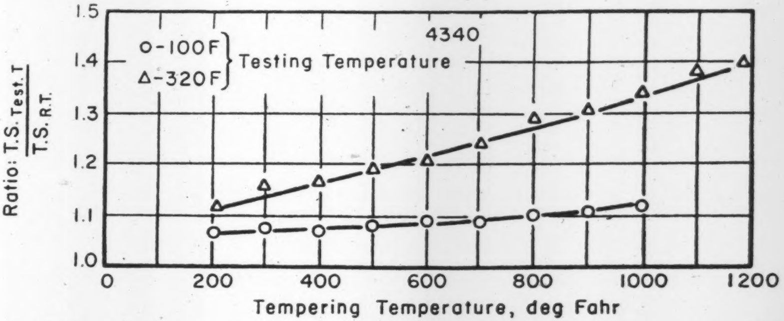

FIG. 6.—Effect of Tempering Temperature on the Low Temperature Strength of 4340 Steel.

General Effects of Increasing Stress Concentrations:

The stress concentration factors for the notch geometry used were obtained from R. E. Peterson's "Stress Concentration Design Factors" (6). The highest values of stress concentration were obtained by calculation and extrapolation in accordance with Appendix A of R. E. Peterson's book. Stress concentrations for notch radii smaller than 0.001 in. are considered accurate only in the order of magnitude.

The effect of a notch on the tensile

strength of heat-treated steels depends upon many factors. Notches having a geometry similar to that investigated here leading to elastic stress concentrations of greater than approximately 2 generally increase by about 50 per cent the strength of steels heat treated to 200,000 psi or less (1). However, as the strength level increases above this value, the notch strength decreases to values that may be very low for very high tensile strength and high stress concentration.

This general trend is also maintained for 4340 steel after the various heat treatments investigated. The results of tests at room temperature, Fig. 7, reveal that the magnitude of stress concentrations has little effect on the notch strength for a tempering temperature of 800 F leading to a tensile strength close to 200,000 psi. However, tempering at lower temperature leads to notch-strength values that generally decrease as the stress concentration increases. The rate of this decrease was found to increase as the tensile strength increased and to be greatest for the lowest tempering temperature generally investigated, namely, 400 F. No indication of the 500 F embrittlement could be observed in the notch tests at room temperature.

At a testing temperature of -100 F, however, the 500 F embrittlement gradually develops, according to Fig. 8, as the stress concentration increases. At the two mildest stress concentrations inves-

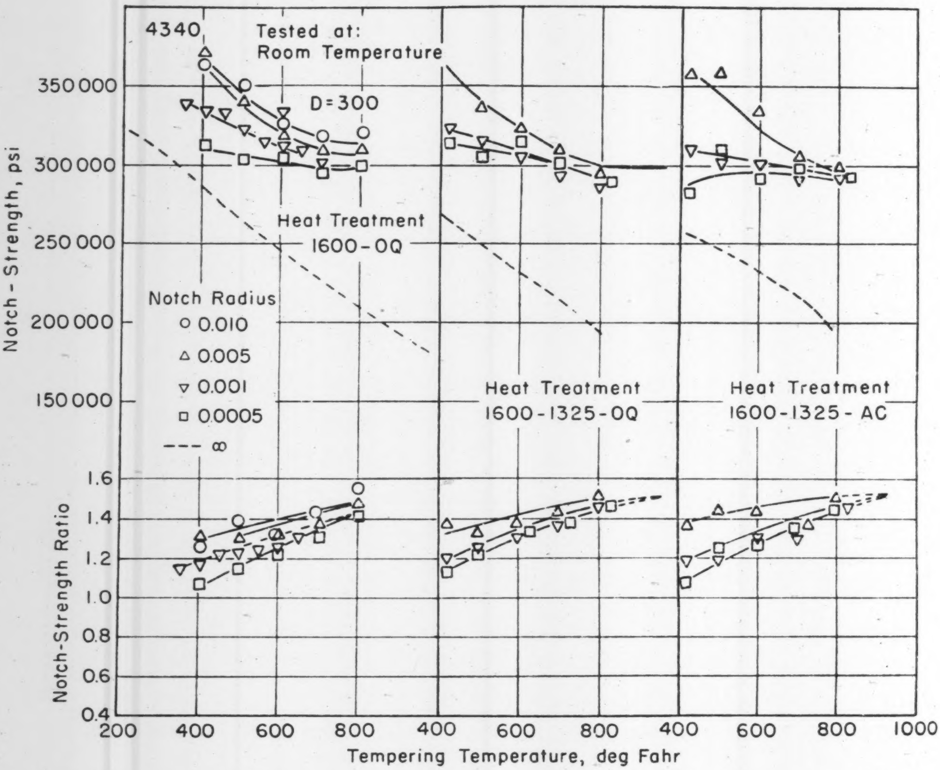

FIG. 7.—Effect of Heat Treatment and Stress Concentration on the Notch Strength and Notch Strength Ratio of 4340 Steel Tested at Room Temperature.

tigated, only a faint indication of this embrittlement could be observed. However, at higher stress concentrations it became distinct for two of the steel conditions investigated, while it was still not definite for the third.

Finally, at −320 F the 500 F embrittlement (Fig. 9) was very pronounced, even at the lowest stress concentrations, and it led to very low notch-strength ratios at the highest stress concentrations.

Cross-plotting the data of Figs. 7 to 9 with stress concentration as abscissa in Fig. 10 further illustrates the progressive embrittlement of the various steel conditions with increasing stress concentration. It is interesting to note that notch radii of less than 0.001 in. caused a small but distinct reduction in notch strength whenever the stress-concentration effect was high. On the other hand, it appears quite definite from these data that thus increasing the stress concentration above a value of approximately 10 leads to a gradual approach to a limiting value of notch strength that is only slightly reduced compared to the value for the stress concentration of 10.

For further comparison of the test results, therefore, notch-strength values for the two highest stress concentrations have been averaged in order to reduce the variations in these data.

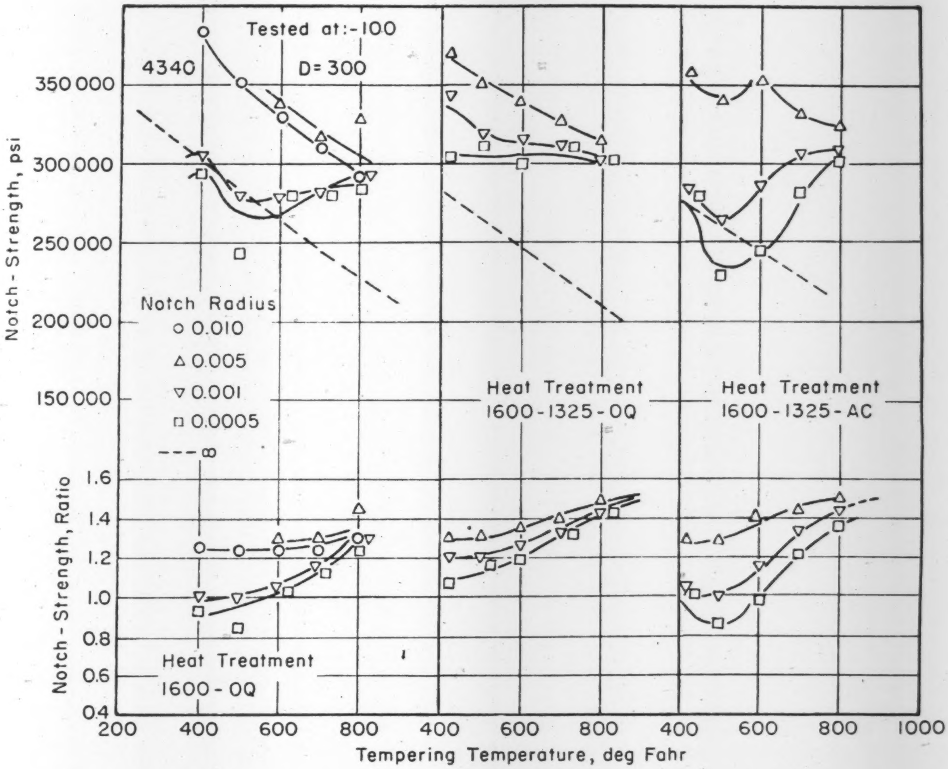

FIG. 8.—Effect of Heat Treatment and Stress Concentration on the Notch Strength and Notch Strength Ratio of 4340 Steel Tested at −100 F.

Effect of Heat-Treating Method on Stress-Concentration Effects:

The heat treatments discussed here, other than the conventional treatment, are aimed at the production of a lower bainite structure on quenching. The ductility of unnotched specimens has been found to be slightly dependent on the heat treating method (Fig. 3). At room temperature, the two-step treatments yielded ductilities that were a few per cent smaller than those of the conventional heat treatments. In addition, the air-cooled specimens exhibited slightly lower ductilities than the two-step oil-quenched specimens at lower tempering temperatures. However, at −320 F these differences became rather small and nearly nil for the higher tempering temperatures. However, this rating of the three heat treatments, derived from ductility values, was not confirmed by the test results on notched specimens.

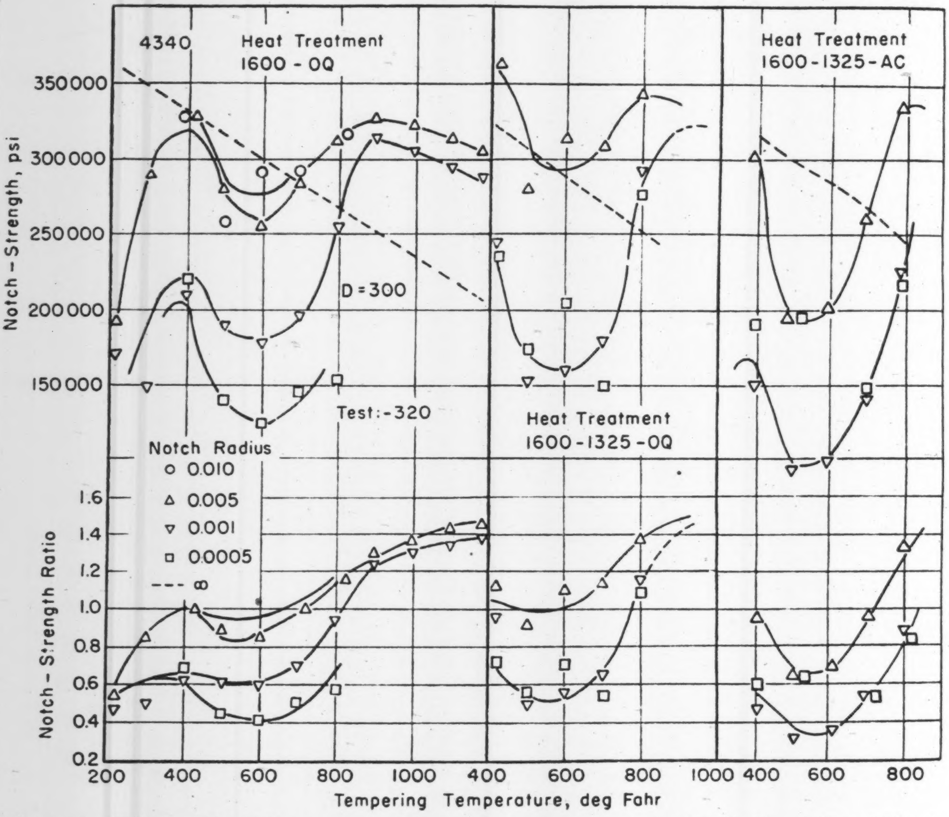

FIG. 9.—Effect of Heat Treatment and Stress Concentration on the Notch Strength and Notch Strength Ratio of 4340 Steel Tested at −320 F.

The notch-strength properties were also found to be different in all three instances, but these relations considerably varied with the tempering temperature, the stress concentration factor, and the testing temperature, as is illustrated in Fig. 10.

At room temperature and a stress-concentration factor of about 4, all three heat treatments yielded the same notch-strength values, within the scattering of the test data. For a stress concentration factor of about 10, the air-cooled material was found to be slightly inferior to the two other heat treatments at the lowest tempering temperatures.

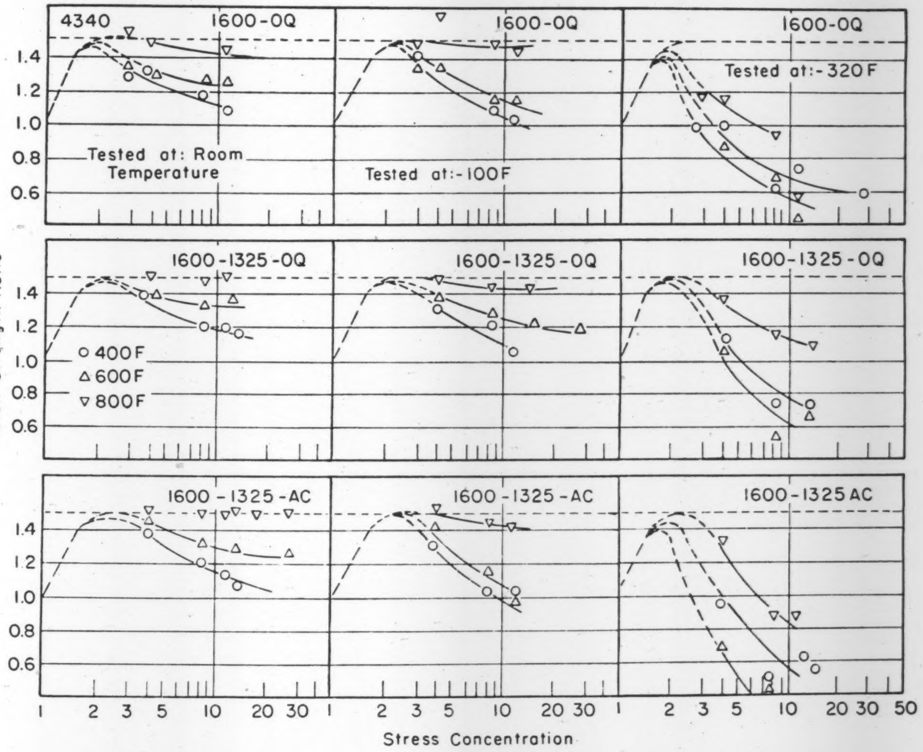

FIG. 10.—Effect of Heat Treatment, Stress Concentration, and Testing Temperature on the Notch Strength Ratio of 4340 Steel.

At −100 F, both two-step treatments yielded slightly higher notch-strength values than the regular oil-quench for the highest tempering temperature. After tempering at from 400 to 600 F, the normal quenched material was intermediate, with the air-cooled condition possessing the lowest notch-strength values. These relations clearly developed as the stress concentration increased.

At −320 F the notch-strength values followed similar relative trends although there was considerable scattering of the test data.

The effects of the various heat treatments are further represented in Fig. 11, where the notch strength is plotted as a function of the tempering temperature. These curves illustrate particularly the superiority of the two-step oil-quenched condition at testing temperatures in the range of −100 F.

The 4340 steel heat treated to strength levels above 200,000 psi has been shown to become increasingly notch sensitive as the notch radius decreases and as the testing temperature is reduced.

At room temperatures, the static notch sensitivity increases gradually as the tensile strength increases above 200,000 psi, and there is no indication of a particularly high notch sensitivity in the range 500 to 700 F.

At a testing temperature of −100 F, similar behavior is observed except that 500 F embrittlement has developed for the most sharply-notched specimens. At −320 F all notched specimens reveal the 500 F embrittlement, to a degree that increases with the sharpness of the notch.

The intensity of the 500 F embrittlement depends further on the heat treating procedure. 4340 steel, if given a two-step heat treatment to suppress the formation of upper bainite (i.e., austenitizing at 1600 F for 1 hr, followed by furnace cooling to 1325 F and holding at that temperature for 1 hr, prior to oil quenching) was found to be less notch sensitive than this steel when given the conventional heat treatment (i.e., oil quenching from 1600 F). However, the structures formed after furnace cooling to 1325 F and subsequent air cooling possess reduced notch toughness even in the absence of obvious slack-quenched products.

The tension tests in this investigation were completed on specimens of small cross-section. Subsequent tests indicate that the notch-properties of the ultra high-strength steels were also a function of the cross-section of the specimen. The effects of as-tested section size on the notch strength of 4340 steel given conventional and two-step heat treatments are now being investigated in the Syracuse University Research Laboratories.

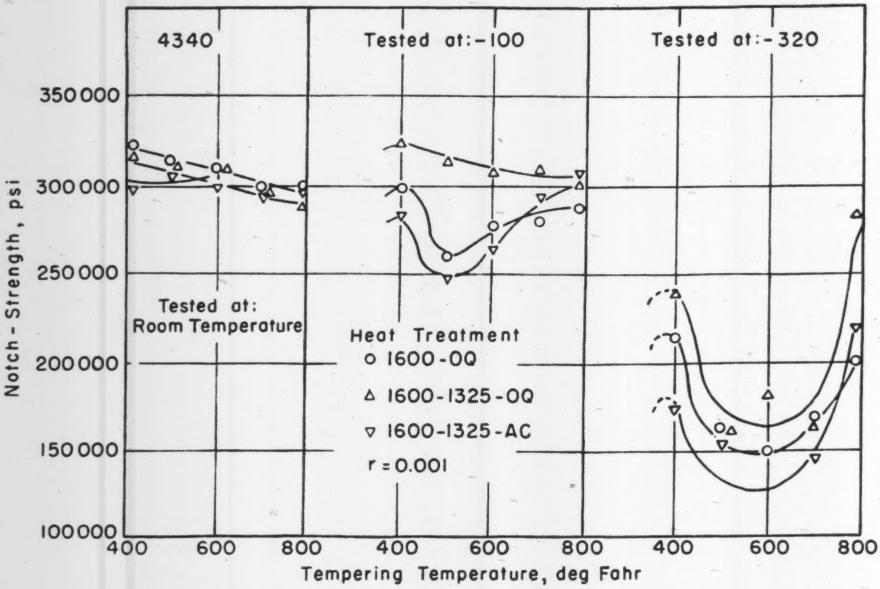

FIG. 11.—Effect of Heat Treatment on the Notch Strength of 4340 Steel Tested at Various Temperatures, as Indicated.

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Much assistance in the execution of the experimentation was provided by T. Rising and J. Cady.

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DISCUSSION

MR. C. M. CARMAN.[1]—From the tone of this paper, it is assumed that 4340 steel is not notch sensitive at room temperature but may be notch sensitive at −320 F. In attempting to interpret these tests in terms of service conditions, does this mean that 4340 steel would be notch sensitive when used in structures or notch sensitive only at low temperatures?

MR. E. P. KLIER (*author*).—The small notch tension specimens indicate that 4340 steel tested at room temperature is notch insensitive, and in service applications where the loaded section is equal to or less than that of these test specimens, the present results should provide a satisfactory index of performance. As the section size increases, the service performance will not be faithfully represented by these results. Under extreme conditions, that is, as the section size increases to large dimensions, notch tensile strengths as given in Fig. 12, may more realistically represent performance.[2]

MR. E. T. WESSEL.[3]—A question arises regarding the preparation of the notched tension specimens used in this investigation. It would be enlightening to know what sort of machining, grinding, or other techniques were used to obtain the very small notch radii (0.0005 to 0.0001 in.) shown in Table I. It appears that very critical machining would be necessary since small deviations in the notch radii would cause relatively large differences in the theoretical stress concentrations for this range of notch sharpness. Any real difference in notch behavior that may exist between these sharp notches would tend to be obscured if such variations of notch radii were present. Such an averaging effect would promote the appearance that, once some critical stress concentration was reached, further increases of stress concentrations (notch sharpness), would have little effect on notch strength as was reported to be the case.

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[1] Metallurgist, Frankford Arsenal, Philadelphia, Pa.

[2] These results depend on the steel composition, heat treatment, notch radius, and specimen size. A paper in which these factors have been more fully examined has been presented to ASTM by E. P. Klier, B. B. Muvdi, and G. Sachs, "Effect of a Number of Variables on the Tensile Properties of High-Strength Steels,"

[3] Research Engineer, Westinghouse Research Laboratory, Pittsburgh, Pa.

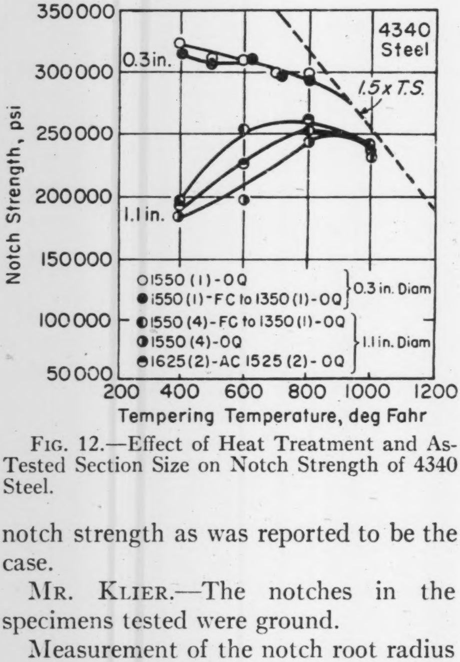

FIG. 12.—Effect of Heat Treatment and As-Tested Section Size on Notch Strength of 4340 Steel.

MR. KLIER.—The notches in the specimens tested were ground.

Measurement of the notch root radius for these very small dimensions is difficult, but it is possible to group the notch radii fairly easily into intervals of 0.00025 in.

None of the specimens was annealed after grinding to relieve grinding stresses. Such stress relief while of possible value for fatigue loading is probably not significant for the present type of test.

MR. OSCAR O. MILLER.[4]—It seems that some other investigator has found that the direct quench from somewhere around 1550 or 1600 F gave them better results than a stepwise treatment. Would the authors like to comment, or do they know what causes this result due to holding at 1325 F.

TABLE II.—AUSTENITIZING SCHEDULES FOR IMPACT STRENGTH STUDY

All Specimens Initially Air Cooled from 1600 F After 1 hr.

A. 1500 F	1 hr - 4 hr - 12 hr - 24 hr
B. 1550 F	1 hr - 4 hr
C. 1600 F	1 hr - 4 hr
D. 1500 F	1 hr, F.C. to 1300 F - 1 hr - 4 hr - 16 hr
E. 1500 F	1 hr, F.C. to 1325 F - 0 hr - 1 hr - 4 hr - 66 hr
F. 1500 F	1 hr, F.C. to 1350 F - 1 hr - 11 hr
G. 1550 F	1 hr, F.C. to 1325 F - 0 hr

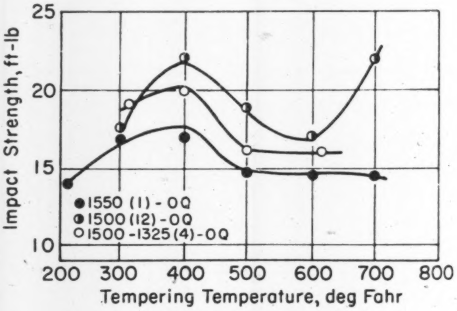

FIG. 13.—The Impact Strength of 4340 Steel after the Indicated Heat Treatments. Points are average of 10 tests.

MR. KLIER.—In addition to the conventional austenitizing treatment and the stepped treatment described in this paper, a third schedule has been proposed. It consists of a high-temperature normalizing followed by a low-temperature austenitizing. The impact strength of 4340 steel after each of these austenitizing treatments has been completed as given in Table II. For certain of these treatments, inferior impact strengths were measured for the stepped austenitizing treatments. The optimum impact strength *versus* tempering temperature data for each of the types of austenitizing schedules are given in Fig. 13. The minimum impact strength *versus* tempering temperature data for each of the schedules are given in Fig. 14. As may be assumed from the cooling rate data in Fig. 2 of the paper and from the measured cooling rates in large sections which have been variously reported,[5] the impact strengths given in Fig. 14 probably more nearly characterize the properties for the structures to be expected in large sections.

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[4] International Nickel Co., Research Laboratory, Bayonne, N. J.

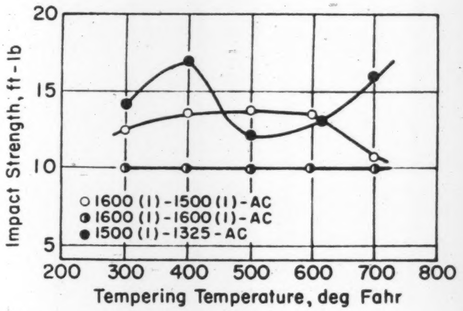

FIG. 14.—The Impact Strength of 4340 Steel after the Indicated Heat Treatments. Points are average of 10 tests.

The impact strength and notch strength may be taken as indexes of notch sensitivity. By taking into consideration that the ductile-brittle ranges as measured in the two tests are separated by a temperature interval as given in Fig. 15, compatible indexes of notch sensitivity for the two test methods are, in principle, possible. However, this is true only when the sections of the service members are of the same dimensions as the test specimens. For large sections the properties to be expected are not faithfully predicted by either of the small scale tests.

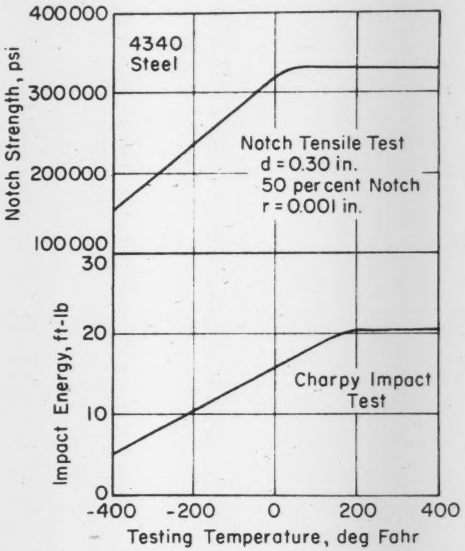

FIG. 15.—Ductile-Brittle Transitions in the Notch-Tensile and Impact Tests as Function of the Testing Temperature.

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[5] For example see Metals Handbook "Quenching Mediums," pp. 615–620, 1948 Ed., Am. Soc. Metals.

EFFECT OF COMPOSITION AND HEAT TREATMENT ON IMPACT PROPERTIES OF AISI TYPES 403 AND 410 STAINLESS STEEL*

By E. J. Whittenberger,[1] E. R. Rosenow,[1] and D. J. Carney[1]

Synopsis

The use of higher carbon contents (0.12 to 0.19 per cent), the addition of nitride- and carbide-forming elements (aluminum, vanadium and titanium), and austenitizing at lower temperatures to achieve finer grain size and minimize intergranular carbide precipitation during subsequent tempering, were found to improve the impact properties of the 12 per cent chromium steels investigated. Increasing amounts of manganese, nickel, and nitrogen did not appear to increase the impact strength.

The most widely used of the martensitic grades of stainless steel are the so-called 12 per cent chromium steels. These steels contain approximately 0.06 to 0.13 per cent carbon and 11.5 to 13.0 per cent chromium. Their resistance to corrosion by many environments and their excellent physical properties at both room and elevated temperatures make them ideally suited for blades and buckets in steam and gas turbines, for valve parts, and for applications in the oil and chemical industries.

In many of these applications, increasing demands for improved performance have prompted the use of these steels at high hardness and impact-strength levels. Therefore, the present investigation was undertaken to determine the most desirable combination of chemical composition, heat treatment, and microstructure that would yield optimum impact strength at high hardness levels.

Previous unpublished investigations at the United States Steel Corp. had indicated that the presence of ferrite in the as-quenched microstructure, obtained by adjustment of the austenitizing temperature, improved the impact strength. Also, the addition of carbon in increasing amounts improved impact properties. However, because the presence of ferrite in as-quenched 12 per cent chromium steels is not considered to be desirable due to its detrimental influence on hot-working characteristics and fatigue properties, further investigation was considered necessary in order to develop a 12 per cent chromium steel, having a minimum of as-quenched ferrite, and optimum impact strength.

Procedure

Twenty six-pound induction-furnace heats of the standard 12 per cent chromium steel composition and 12 per cent chromium steels modified with carbon, manganese, nickel, vanadium, titanium, aluminum, and nitrogen were melted to study the relative effect of these additions on the Charpy and Izod impact properties. Also included in this program was one commercial 12 per cent chromium steel with erratic impact values in heat-treated bars.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] Chief Development Metallurgist, Physicist, and Superintendent, No. 2 Electric Furnace Shop, respectively, South Works, United States Steel Corporation, Chicago, Ill.

TABLE I.—CHEMICAL ANALYSES OF THE 12-PER-CENT CHROMIUM STEELS INVESTIGATED.

Code	Series	Carbon, per cent	Manganese, per cent	Phosphorus, per cent	Sulfur, per cent	Silicon, per cent	Nickel, per cent	Chromium, per cent	Nitrogen, per cent	Aluminum, per cent	Vanadium, per cent	Titanium per cent
No. 1	Standard	0.06	0.53	0.007	0.015	0.32	0.11	12.50	0.041	...	...	...
No. 2	Standard	0.12	0.53	0.008	0.015	0.43	0.09	12.74	0.038	...	...	...
No. 3	Standard	0.17	0.50	0.012	0.036	0.33	0.21	12.04	0.025	...	...	...
No. 4	Standard	0.19	0.61	0.008	0.034	0.35	0.20	12.65	0.021	...	...	...
No. 5	Manganese	0.08	1.04	0.008	0.018	0.27	0.12	13.10	0.047	...	...	...
No. 6	Manganese	0.11	1.02	0.009	0.030	0.36	0.21	12.52	0.028	...	...	...
No. 7	Manganese	0.14	1.08	0.009	0.028	0.32	0.09	12.08	0.029	...	...	...
No. 8	Manganese	0.19	0.96	0.010	0.032	0.34	0.11	12.42	0.036	...	...	...
No. 9	Manganese	0.07	1.36	0.012	0.031	0.37	0.22	12.22	0.035	...	...	...
No. 10	Manganese	0.11	1.44	0.011	0.033	0.34	0.13	12.36	0.031	...	...	...
No. 11	Manganese	0.14	1.46	0.010	0.030	0.35	0.13	12.26	0.057	...	...	...
No. 12	Manganese	0.18	1.44	0.011	0.030	0.37	0.12	12.18	0.039	...	...	...
No. 13	Nickel	0.08	0.44	0.011	0.042	0.40	0.59	12.22	0.034	...	...	...
No. 14	Nickel	0.12	0.46	0.010	0.039	0.46	0.60	12.38	0.036	...	...	...
No. 15	Nickel	0.15	0.36	0.009	0.034	0.35	0.50	12.60	0.028	...	...	...
No. 16	Nickel	0.19	0.49	0.009	0.042	0.44	0.52	12.48	0.070	...	...	...
No. 17	Nickel	0.09	0.46	0.009	0.042	0.47	1.02	12.32	0.036	...	...	...
No. 18	Nickel	0.12	0.68	0.010	0.035	0.43	1.15	11.92	0.032	...	...	...
No. 19	Nickel	0.16	0.44	0.011	0.031	0.44	1.04	11.96	0.038	...	...	...
No. 20	Nickel	0.19	0.67	0.008	0.052	0.31	1.03	12.48	0.024	...	...	...
No. 21	Nitrogen	0.08	0.41	0.008	0.009	0.29	0.08	12.94	0.13	...	...	...
No. 22	Nitrogen	0.12	0.41	0.009	0.013	0.19	0.08	12.00	0.12	...	...	...
No. 23	Nitrogen	0.17	0.40	0.009	0.012	0.27	0.05	12.32	0.11	...	...	...
No. 24	Nitrogen	0.21	0.39	0.007	0.020	0.25	0.10	11.85	0.12	...	...	...
No. 25	Nitrogen	0.06	0.49	0.009	0.026	0.52	0.04	11.71	0.060	...	...	...
No. 26	Nitrogen	0.10	0.51	0.010	0.022	0.47	0.07	11.75	0.098	...	...	...
No. 27	Manganese	0.09	0.92	0.007	0.034	0.46	0.05	12.18	0.023	...	...	...
No. 28	Standard	0.10	0.46	0.010	0.027	0.34	0.12	11.57	0.048	...	...	...
No. 29	Aluminum	0.10	0.49	0.010	0.022	0.37	0.14	11.51	0.069	0.09	...	...
No. 30	Vanadium	0.10	0.54	0.010	0.025	0.34	0.08	12.28	0.023	0.015	0.07	...
No. 31	Titanium and Aluminum	0.10	0.48	0.011	0.024	0.38	0.09	11.72	0.016	0.04	...	0.07
No. 32	Commercial	0.10	0.45	0.019	0.010	0.26	0.20	12.18	...	...	...	...
...	Bar 14	0.09	0.46	0.025	0.011	0.31	0.24	12.24	...	...	...	...
...	Bar 6	0.10	0.44	0.023	0.013	0.24	0.24	12.24	...	...	...	...

The laboratory induction-furnace heats were cast in graphite molds $2\frac{1}{2}$ in. in diameter by 14 in. high and subsequently forged to $1\frac{1}{4}$ in. diameter and $\frac{5}{8}$ in. square bars for tension and impact specimens, respectively. Pilot specimens, $\frac{5}{8}$-in. cubes, were secured from each heat to determine the as-quenched hardness for various austenitizing temperatures.

TABLE II.—EFFECT OF CARBON, MANGANESE, NICKEL, AND NITROGEN CONTENTS UPON THE MECHANICAL PROPERTIES AND MICROSTRUCTURE OF 12 PER-CENT-CHROMIUM STEELS TEMPERED TO SEVERAL HARDNESS LEVELS.

Code	Identifying Elements, per cent	Austenitizing Temperature, deg Fahr[a]	Brinell Hardness After Oil Quenching	Tempering Temperature, deg Fahr[a]	Brinell Hardness After Tempering	V-Notch Charpy Impact Results, ft-lb				Ferrite Rating	ASTM Grain Size
						—40 F	0 F[b]	80 F[b]	120 F		
No. 1......	0.06 C 0.041 N	1750	415	1025	321	...	11– 8– 8 (9)	19–30–30 (26)	20–36–41	Trace	5
No. 2......	0.12 C 0.038 N	1750	444	1050	285	6– 9–11	13–18–20 (17)	38–41–45 (41)	...	Trace	6
No. 3......	0.17 C 0.025 N	1750	461	1050	302	22–26–28	24–26–31 (27)	36–37–42 (38)	...	Trace	6
No. 4......	0.19 C 0.021 N	1750	514	1075	285	11–24–28	32–34–40 (35)	72–77–82 (77)	...	Trace	7
No. 5......	0.08 C 1.04 Mn	1750	477	1050	302	6– 7–10	8–16–19 (14)	12–30–34 (25)	...	1 per cent	7
No. 6......	0.11 C 1.02 Mn	1750	477	1050	285	18–22–26	25–26–36 (29)	26–32–32 (30)	...	Trace	6
No. 7......	0.14 C 1.08 Mn	1750	444	1050	285	7–13–16	8–20–26 (18)	24–26–27 (26)	...	None	6
No. 8......	0.19 C 0.96 Mn	1750	514	1075	302	7–13–23	6–12–24 (14)	22–30–36 (29)	...	None	8
No. 9......	0.07 C 1.36 Mn	1750	401	1025	302	...	6– 7–13 (9)	9–13–22 (15)	18–24–26	None	7
No. 10......	0.11 C 1.44 Mn	1750	444	1025	302	...	4– 9–13 (9)	6– 8–23 (12)	7–14–24	None	5
No. 11......	0.14 C 1.46 Mn	1750	495	1075	311	...	2– 6– 7 (5)	15–16–22 (17)	10–24–28	None	6
No. 12......	0.18 C 1.44 Mn	1750	514	1075	302	10–10–24	14–20–24 (19)	19–19–22 (20)	...	None	5
No. 13......	0.08 C 0.59 Ni	1750	401	1025	311	5– 7– 8	6–14–18 (13)	29–30–30 (29)	...	1 per cent	6
No. 14......	0.12 C 0.60 Ni	1750	444	1025	321	...	9–12–12 (11)	20–28–28 (25)	27–28–33	None	6
No. 15......	0.15 C 0.50 Ni	1750	444	1075	285	25–26–28	30–32–34 (32)	56–60–65 (60)	...	None	6
No. 16......	0.19 C 0.52 Ni	1750	514	1100	311	21–24	10–22–24 (19)	28–31–32 (30)	...	None	7
No. 17......	0.09 C 1.02 Ni	1750	444	1025	311	11–13–13	8–20–20 (16)	4–24–28 (18)	...	None	7
No. 18......	0.12 C 1.15 Ni	1750	461	1025	311	7–11–13	9–11–22 (14)	24–25–25 (25)	...	None	6
No. 19......	0.16 C 1.04 Ni	1750	495	1050	302	20–22–22	20–22–25 (22)	26–28–28 (27)	...	None	6
No. 20......	0.19 C 1.03 Ni	1750	555	1075	293	13–21–22	16–18–18 (17)	22–22–24 (23)	...	None	6
No. 21......	0.08 C 0.13 N	1750	477	1025	293	...	3– 4– 6 (4)	4– 4– 6 (5)	...	None	7
No. 22......	0.12 C 0.12 N	1750	534	1100	311	10–21–25	18–22–25 (22)	30–32–32 (31)	...	None	5
No. 23......	0.17 C 0.11 N	1750	578	1125	302	18–20–30	9–22–24 (18)	44–48–52 (48)	...	None	7
No. 24......	0.21 C 0.12 N	1750	601	1175	285	10–14–16	25–26–28 (26)	26–48–60 (45)	...	None	7

[a] Held 1 hr at temperature.

[b] Designates averages.

To determine the optimum austenitizing temperature, $\frac{5}{8}$-in. cube pilot specimens of the heats with four carbon levels and varying manganese, nickel, and nitrogen contents (Codes 1 to 24, Table I) were austenitized at temperatures from 1700 to 1850 F at 50 degree intervals. Specimens were held at temperature for 45 min and oil quenched. The results of the hardness measurements of these specimens suggested that an austenitizing temperature of 1750 F would be satisfactory for all the compositions involved. This temperature was in agreement with the commercial heat-treating temperature range of 1700 to 1850 F. Within this temperature range, the austenite field for 12 per cent chromium steel is the broadest.

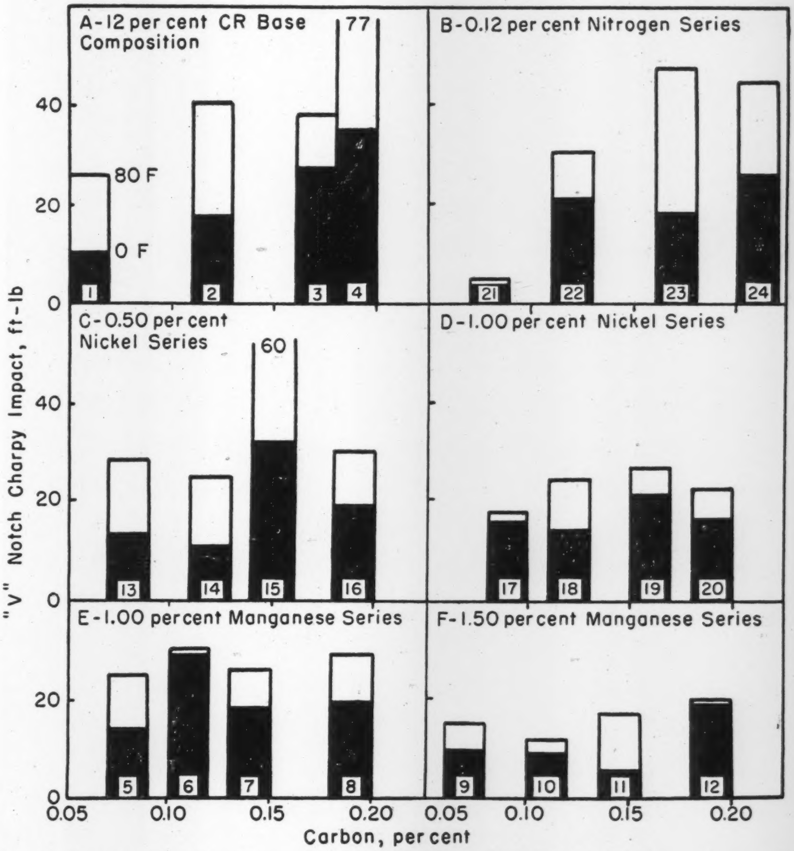


FIG. 1.—Influence of Carbon Content Upon the 0 and 80 F V-Notch Charpy Impact Strengths of The Modified 12 per cent Chromium Steels after Austenitizing at 1750 F Followed by Oil Quenching and Tempering to 300 Brinell Hardness Number. Number on Bar is the Coded Heat Number (Table I).

Test coupons for impact specimens were austenitized at 1750 F for 1 hr and oil quenched, then drawn for 1 hr at a temperature to obtain a Brinell hardness number of 300 or greater. Tempering to this high hardness level was done so that a critical evaluation of these experimental compositions could be made. V-notch Charpy impact specimens were machined and tested at 0 and 80 F. If the results obtained at 0 F were above 10 ft-lb, a third testing temperature of −40 F was used. If the impact results were below 10 ft-lb at 0 F, a third testing temperature of +120 F was used. The temperatures at which the impact tests were made were considered to be within ±5 F of the indicated temperature. In addition, tensile properties and keyhole Charpy impact strengths were determined for the steels representing the 0.12, 0.17, and 0.19 per cent carbon levels of the base 12 per cent chromium compositions heat treated as outlined above.

For the portion of the investigation pertaining to the effect of microstructure on impact strength of the 0.10 per cent carbon steel, specimens from a commercial heat (Code 32) and laboratory heats containing additions of aluminum, vanadium, and a titanium-zirconium-aluminum agent were obtained. To determine the extent of austenite formation, $\frac{5}{8}$-in. cube pilot specimens of these steels were austenitized at temperatures from 1500 to 1950 F at 50-degree intervals for 1 hr and quenched in oil. Hardness readings and metallographic observations were made on each specimen. The lowest austenitizing temperature that produced the maximum hardness in each steel was then selected as the quenching temperature for tension and impact testing. The test coupons were tempered to several hardness levels and keyhole Charpy impact properties were determined. Metallographic studies of grain size and carbide distribution were made on each of the impact specimens tested.

RESULTS AND DISCUSSION

Influence of Chemical Composition on Impact Strength:

The results of the impact testing and metallographic observations are presented in Table II. The impact results obtained at 0 and 80 F are summarized in Fig. 1.

In analyzing the impact results, consideration was given to the effect of a given element on the level of impact strength as well as its effect on the temperature range over which the impact strength was 15 ft-lb or above. As will be noted in Table II, carbon has a marked effect on improving the impact strength level at 0 and 80 F, thus broadening the temperature range for good impact behavior in the 12 per cent chromium-base composition. Higher carbon contents also improve impact characteristics of the 12 per cent chromium steels containing 0.12 per cent nitrogen, 0.5 nickel, 1.0 nickel, and 1.5 manganese. The effect of carbon on 12 per cent chromium steels containing 1.0 per cent manganese was not consistent.

In comparison with carbon, the effect of the other elements—, nickel, manganese, and nitrogen—, was minor. Indeed, with the exception of 1 per cent manganese and 0.50 and 1 per cent nickel, both of which improved the impact characteristics of the low-carbon 12-per cent chromium steels at 0 F, addition of these elements had no effect or were detrimental to impact characteristics.

Because of the favorable improvement in the V-notch Charpy impact properties of the three higher carbon levels of the base composition, keyhole Charpy and Izod impact properties were also determined. Samples were austenitized at 1750 F and were tempered to Brinell hardnesses of approximately 300 for keyhole Charpy testing and approximately 230 for Izod impact testing. The impact results are listed in Table III. All three carbon levels (0.12, 0.17, and 0.19 per cent) of the base composition Codes 2, 3, and 4, exhibited encouraging keyhole Charpy impact properties, 35 ft-lb or better, and good Izod impact

TABLE III.—EFFECT OF CARBON CONTENT AND HEAT TREATMENT UPON THE MECHANICAL PROPERTIES AND MICROSTRUCTURE OF 12 PER-CENT-CHROMIUM STEELS.

Code	Identifying Elements, per cent	Austenitizing Temperature, deg Fahr[a]	Brinell Hardness after Oil Quenching	Tempering Temperature, deg Fahr[a]	Brinell Hardness after Tempering	Tensile Strength, psi	Yield Strength, psi	Elongation in 2 in., per cent	Reduction of Area, per cent	Keyhole-Charpy Impact Results, ft-lb at 80 F	V-Notch-Charpy Impact Results, ft-lb at 80 F	Izod Impact Results, ft-lb at 80 F	Ferrite Rating, per cent	ASTM Grain Size
No. 2....	0.12 C base	1750	444	1030 to	285	139 850[b]	114 900	17.5	64.0	38–42–48 (42)[d]	38–41–45 (41)[d]		Trace	6
				1115	229	112 900[c]	75 900	23.5	68.4			88–92–98 (93)[d]		
No. 3....	0.17 C base	1750	461	1030 to	302	141 850[b]	113 900	18.5	61.3	34–34–36 (35)	36–37–42 (38)		Trace	6
				1180	229	113 400[c]	73 400	22.0	66.3			80–82–88 (83)		
No. 4....	0.19 C base	1750	514	1030 to	302	141 850[b]	114 000	18.0	61.3	36–36–38 (37)	72–77–82 (77)		Trace	7
				1200	229	112 000[c]	71 430	23.0	65.4			80–80–82 (80)		

[a] Held 1 hr at temperature.
[b] Tempered to 260/320 BHN.
[c] Tempered to 200/240 BHN.
[d] Designates averages.

TABLE IV.—THE EFFECT OF COMPOSITION AND HEAT TREATMENT UPON THE MECHANICAL PROPERTIES AND MICROSTRUCTURE OF 12 PER-CENT-CHROMIUM STEELS.

Code	Identifying Elements, per cent	Austenitizing Temperature, deg Fahr[a]	Brinell Hardness after Oil Quenching	Tempering Temperature, deg Fahr[a]	Brinell Hardness after Tempering	Tensile Strength, psi	Yield Strength, psi	Elongation in 2 in., per cent	Reduction of Area, per cent	Keyhole-Charpy Impact Results ft-lb at 80 F	V-Notch Charpy Impact Results ft-lb at 80 F	Izod Impact Results ft-lb at 80 F	Ferrite Rating, per cent	ASTM Grain Size
No. 6...	0.11 C 1.02 Mn	1650	444	1000	285	[b]	...	...	...	36–34–38 (36)[d]	30–36–46 (38)[d]	...[d]	1	6
No. 10...	0.11 C 1.44 Mn	1625	444	1000	277	[b]	...	...	...	28–28–34 (30)	10–12–12 (12)	...	1 to 2	7
No. 14...	0.12 C 0.60 Ni	1650	444	1000	277	[b]	...	...	...	40–38–38 (38)	38–38–46 (40)	...	1	8
No. 18...	0.12 C 1.15 Ni	1625	444	1000	302	[b]	...	...	...	32–32–42 (35)	32–32–40 (34)	...	1	8
No. 25...	0.06 C 0.06 N	1650	388	1050	285	135 350[b]	119 000	19.5	67.0	30–34–29 (31)	...		None	7
		1650	388	1160	235	107 200[c]	76 930	17.0	56.0			90–82–82 (84)		
		1750	388	1050	302	[b]				6–12–22 (13)	...		None	5
No. 26...	0.10 C 0.09 N	1650	444	1075	285	141 350[b]	120 400	15.5	50.0	32–38–34 (35)	...		None	7.5
		1650	444	1160	235	117 900[c]	79 930	17.0	...			76–74–76 (75)		
		1750	461	1050	352	[b]				6– 8– (7)	...		None	4
No. 27...	0.09 C 0.92 Mn	1650	388	1025	269	132 400[b]	116 400	22.5	68.8	40-42-43 (42)	...		3	6
		1650	375	1160	235	109 900[c]	74 430	22.0	68.8			80-78-78 (78)		
		1750	415	1050	285	[b]				32-31-34 (32)	...		2 to 3	5.5
No. 28...	0.10 C base	1650	415	1050	302	135 350[b]	118 900	19.5	65.4	32-34-34 (33)	...		None	6
		1650	401	1160	235	106 100[c]	73 430	23.0	69.3			84–80–86 (83)		
		1750	429	1050	302	[b]				16–22–28 (20)	...		None	4
No. 29...	0.10 C 0.09 Al	1650	415	1050	302	137 850[b]	121 400	19.0	64.7	30–34–36 (34)	...		None	<8
		1650	415	1160	235	110 400[c]	76 430	22.5	67.5			92–80–82 (84)		
		1750	429	1050	302	[b]				30–32–28 (30)	...		None	<8
No. 30...	0.10 C 0.07 V	1650	388	1025	269	138 500[b]	122 900	18.0	65.4	40–42–44 (42)	...		<1	<8
		1650	415	1160	235	114 900[c]	82 920	19.5	67.7			94–75–84 (84)		
		1750	388	1050	302	[b]				32–34–36 (34)	...		<1	<8
No. 31...	0.10 C 0.07 Ti	1650	375	975	321	154 800[b]	136 500	17.0	62.8					
	and 0.04 Al	1650	375	985	302					34–36–38 (36)	...		2 to 3	<8
		1750	388	1000	285					36–28–35 (33)	...		None	6

[a] Held 1 hr at temperature.

[b] Tempered to 260 to 320 Brinell hardness.

[c] Tempered to 200 to 240 Brinell hardness.

[d] Designates averages.

strengths, 93, 83, and 80 ft-lb, respectively. Each heat contained a small amount of ferrite which influenced the good impact results obtained on these steels.

Effect of Heat Treatment and Microstructure on Impact Properties:

In the study of the effect of microstructure, emphasis was placed upon the effect of prior austenitic grain size and amount and distribution of carbides in a steel essentially free of ferrite. The grain size was varied by a variation of the austenitizing temperature, and the amount and distribution of carbides were varied by addition of carbide- or nitride-forming elements. Included in this portion of the investigation were the 0.12

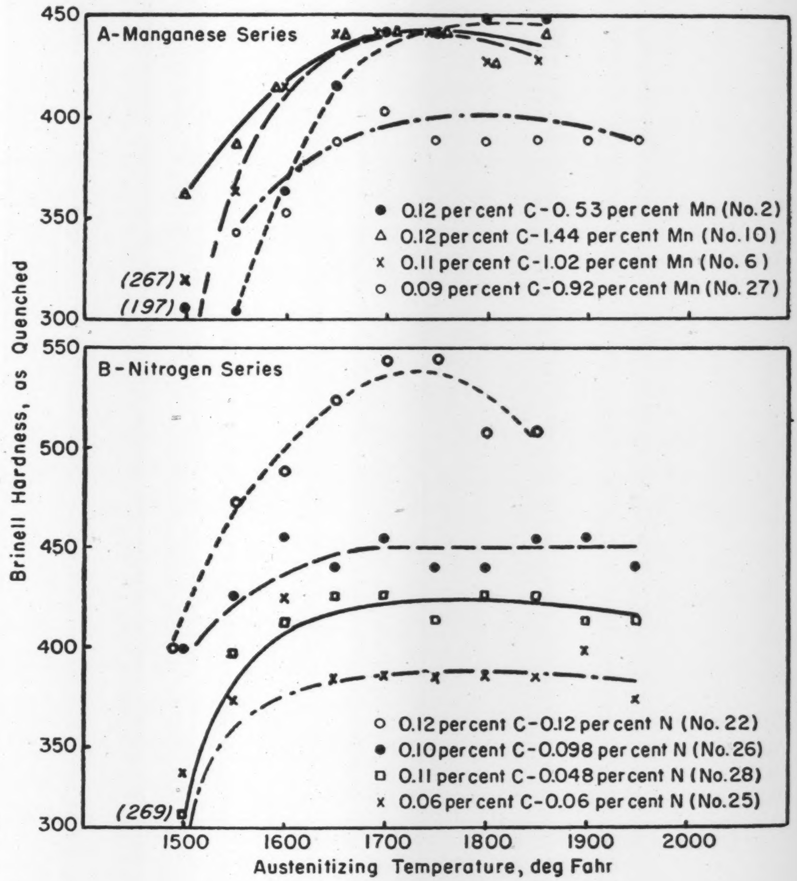


FIG. 2.—Effect of Austenitizing Temperature on the Hardness of 12 per cent Chromium Steels Oil Quenched After 1 hr at Temperature.

In summary of the effect of chemical composition, increasing the carbon content increased the impact strength of the base composition and of several of the modified compositions studied. Increasing the manganese, nickel, and nitrogen contents did not produce any material improvement in impact properties.

per cent carbon, manganese, and nickel series from the first part of the investigation (Codes 6, 10, 14, and 18) and additional laboratory heats containing 0.10 per cent carbon with small amounts of aluminum, vanadium, and a titanium-zirconium-aluminum addition agent.

as a function of the temperature in Figs. 2 and 3. The ferrite and grain size ratings for the heat treatments chosen for impact testing are listed in Table IV.

Hardness results obtained from the quenched specimens were used to de-

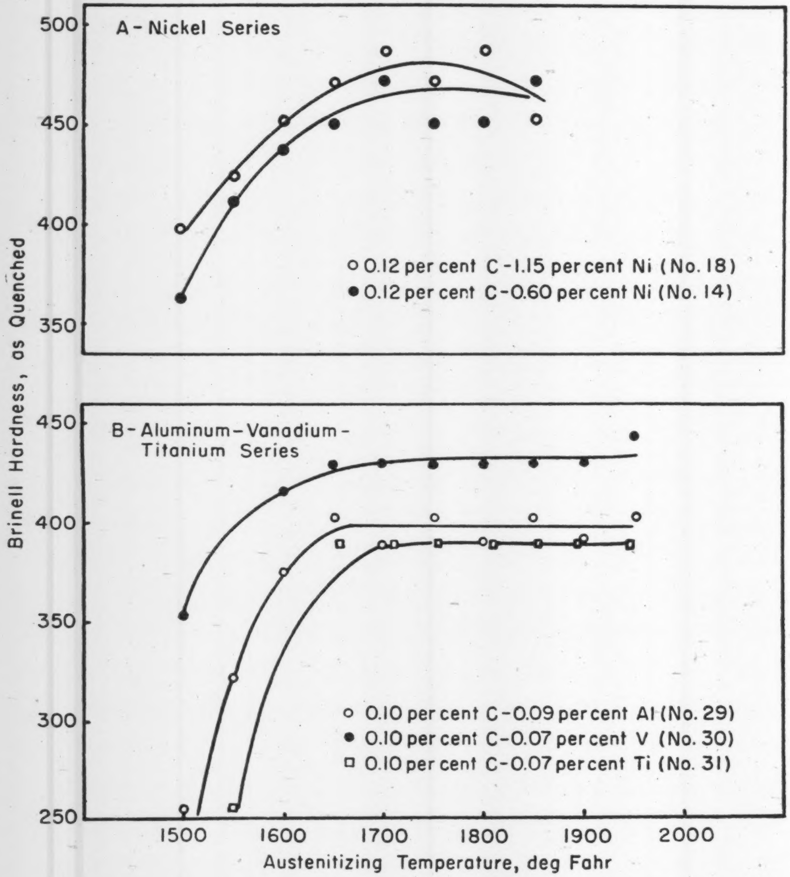


FIG. 3.—Effect of Austenitizing Temperature on the Hardness of 12 per cent Chromium Steels Oil Quenched After 1 hr at Temperature.

To determine the relationship between the austenitizing temperature and the extent of ferrite precipitation for these heats (Codes 6, 10, 14, 18, and 25 to 32, Table I), $\frac{5}{8}$-in. cube specimens were heated for 1 hr at the temperatures selected for the study and then oil quenched. Hardness results are plotted

termine the minimum austenitizing temperature that would produce a completely austenitic structure, except for undissolved carbides. For the majority of the steels studied, an austenitizing temperature of 1650 or 1625 F produced a structure practically free of ferrite, with varying amounts of undissolved carbides or nitrides. A few of the steels tested contained very small amounts of ferrite (less than 1 per cent) at this temperature (Table IV). The hardness results obtained for the various steels varied with the chemical composition and, in general, increased with the carbon content. Increasing the percentage of manganese or nickel had little effect on hardness over the range of temperatures investigated. The lower maximum hardness of 0.09 carbon—0.92 manganese heat (No. 27 in Fig. 2(*a*)) was caused by the lower carbon content and the presence of a small amount of ferrite in the quenched structure (Table IV).

As shown in Fig. 3(*b*), the hardness results for the heat with aluminum were higher than the hardness obtained in the comparable base composition. The addition of vanadium and titanium resulted in a small decrease in the quenched hardness (Fig. 3(*b*)), as would be expected with the presence of undissolved carbides.

Tension and Charpy and Izod impact specimens for the above studies were heat treated at 1650 or 1625 F and tempered to a Brinell hardness of approximately 300 for keyhole Charpy impact testing and approximately 230 for Izod impact testing. In order to evaluate the effect of grain size and undissolved carbides on the impact strength, additional keyhole Charpy impact specimens from several heats (Codes 25 to 31) were austenitized at 1750 F and were subsequently tempered for comparison with the specimens austenitized at 1650 F. The results of the testing at both austenitizing temperatures are listed in Table IV.

For comparison with data obtained in the first portion of this investigation, V-notch Charpy impact tests were made

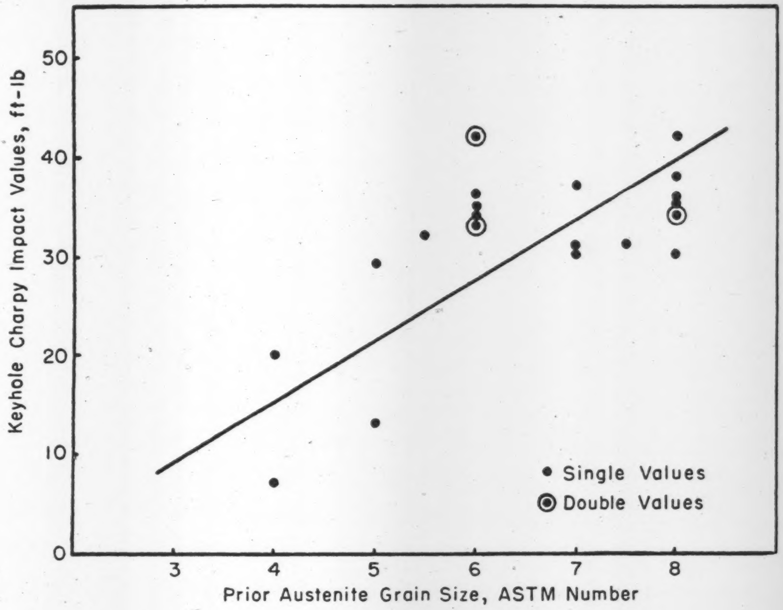
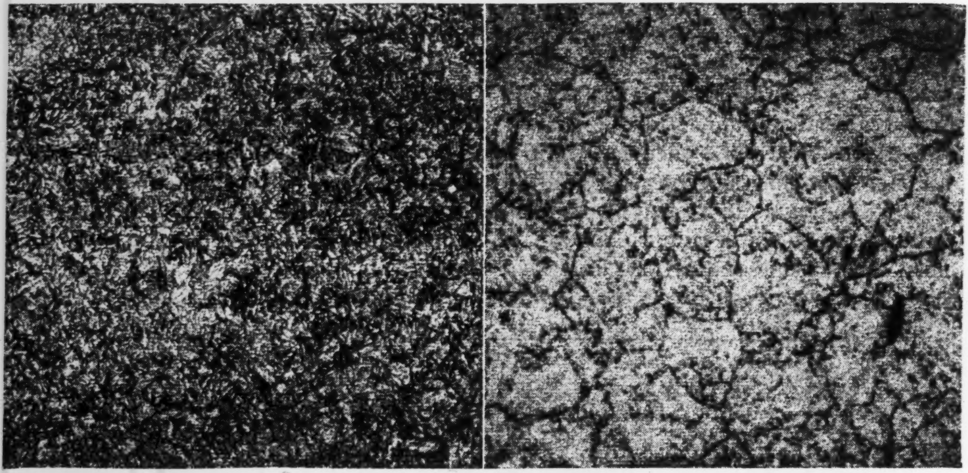
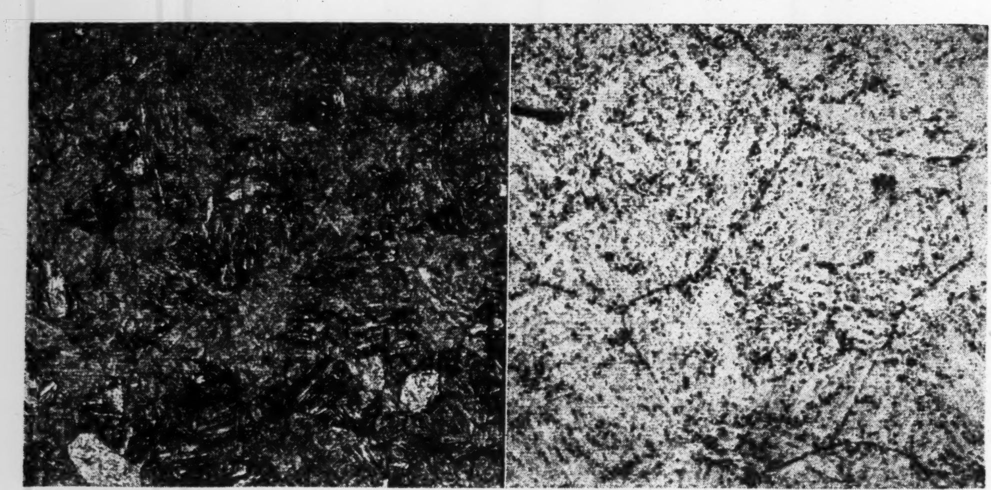


FIG. 4.—Relationship of the Prior Austenite Grain Size and the Room-Temperature Keyhole Charpy Impact Values for Various 12 per cent Chromium Steels Investigated. Each data point represents the average of three test results for a specific composition and heat treatment.



(*a*) Prior austenite grain size No. 6 ASTM rating. (× 100).

(*b*) Carbide precipitate within grains and a discontinuous carbide grain boundary network. Electrolytic chromic acid etch (× 500).



(*c*) Prior austenite grain size No. 4 ASTM rating. Picric-HCl acid etch (× 100).

(*d*) Same as specimen (*b*) except a continuous carbide grain boundary network. Electrolytic sodium cyanide etch (× 500).

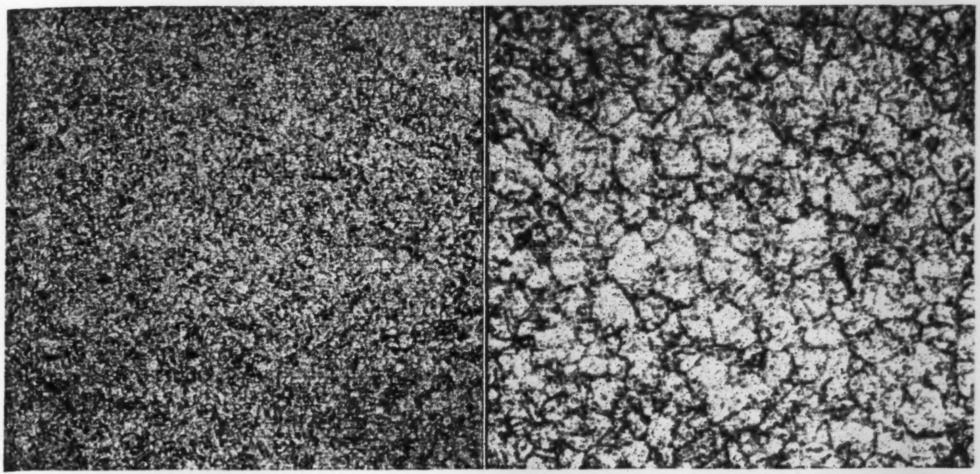
FIG. 5.—Comparison of the Microstructure of a Quenched and Tempered 12 per cent Chromium Steel (No. 28) of the Base Composition.

(*a*) and (*b*)—Oil quenched from 1650 F, drawn at 1050 F.
(*c*) and (*d*)—Oil quenched from 1750 F, drawn at 1050 F.

on specimens from heat Nos. 6, 10, 14, and 18 heat treated to the same hardness level as the Charpy keyhole specimens.

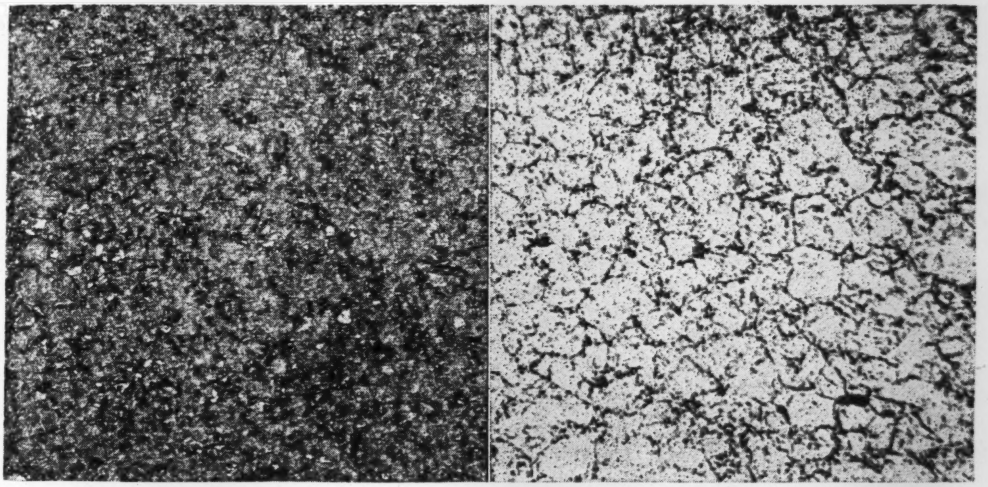
The interpretation of the results listed in Table IV was rendered difficult because the separate effects of carbide distribution and grain size could not be completely isolated. However, some generalization can be made.

A decrease in grain size which was



(*a*) Prior austenite grain size number finer than 8 ASTM rating. (× 100).

(*b*) Carbide precipitate within grains and a discontinuous carbide grain boundary network. Electrolytic chromic acid etch (× 500).

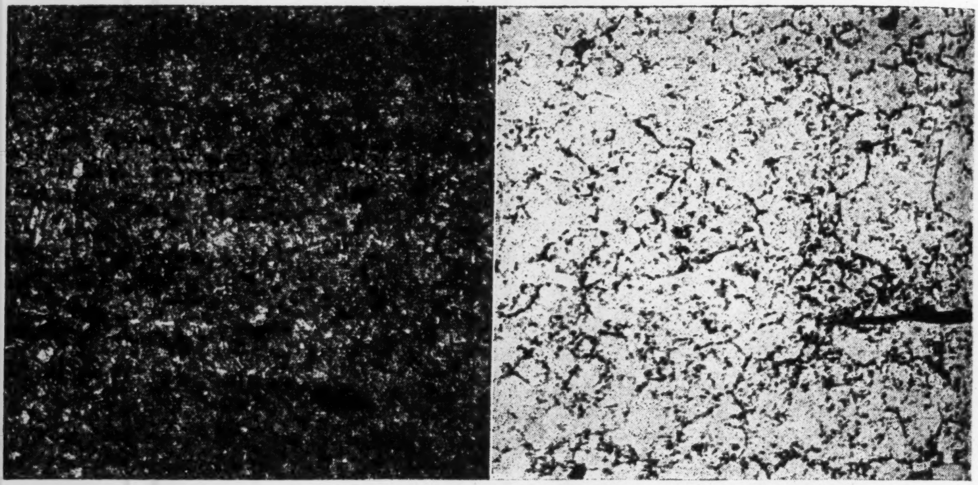


(*c*) Prior austenite grain size number finer than 8 ASTM rating. Picric-HCl acid etch (× 100).

(*d*) Same as specimen (*b*) except less carbide precipitate within the grain. Electrolytic sodium cyanide etch (× 500).

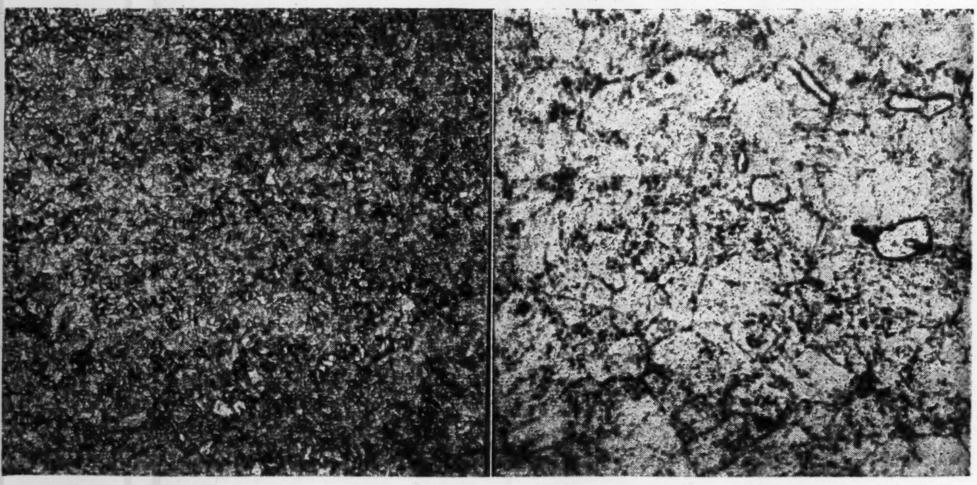
FIG. 6.—Comparison of the Microstructure of a Quenched and Tempered 12 per cent Chromium Steel (No. 29) of the Base Composition Plus 2 lb Aluminum Per Ton.
(*a*) and (*b*)—Oil quenched from 1650 F, drawn at 1050 F.
(*c*) and (*d*)—Oil quenched from 1750 F, drawn at 1050 F.

accomplished by a decrease in austenitizing temperature or by the addition of aluminum, vanadium, or titanium plus aluminum always resulted in an improvement in impact strength. This effect of grain size is illustrated by Fig. 4, in which grain size is plotted against impact strength for all steels regardless of their chemical composition or carbide distribution. As will be noted, an average



(*a*) Prior austenite grain size finer than No. 8 ASTM rating. (× 100).

(*b*) Delta ferrite and carbide precipitate within grains and a trace of carbide grain boundary network. (× 500).



(*c*) Prior austenite grain size finer than No. 8 ASTM rating. Picric-HCl acid etch (× 100).

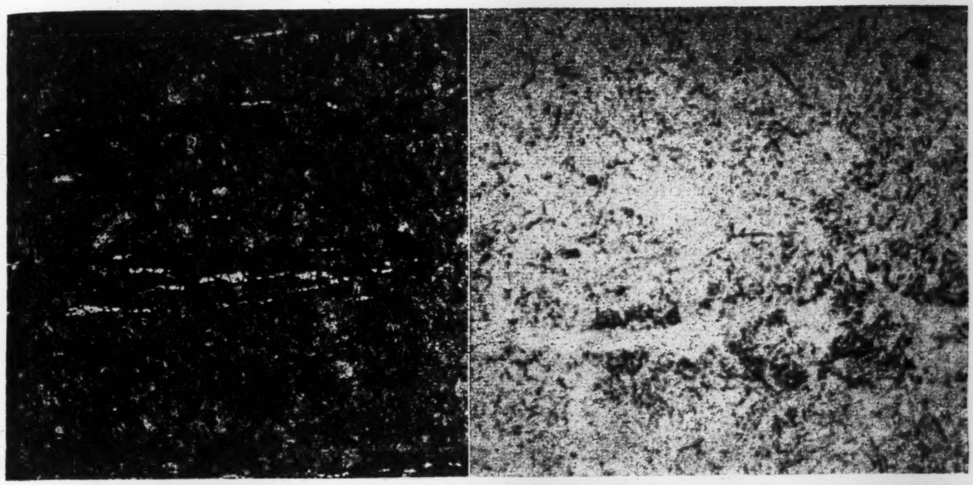
(*d*) Same as specimen (*b*) except more carbide grain boundary network. Electrolytic chromic acid etch (× 500).

Fig. 7.—Comparsion of the Microstructure of a Quenched and Tempered 12 per cent Chromium Steel (No. 30) Of the Base Composition Plus 0.07 per cent Vanadium.

(*a*) and (*b*)—Oil quenched from 1650 F, drawn at 1050 F.
(*c*) and (*d*)—Oil quenched from 1750 F, drawn at 1050 F.

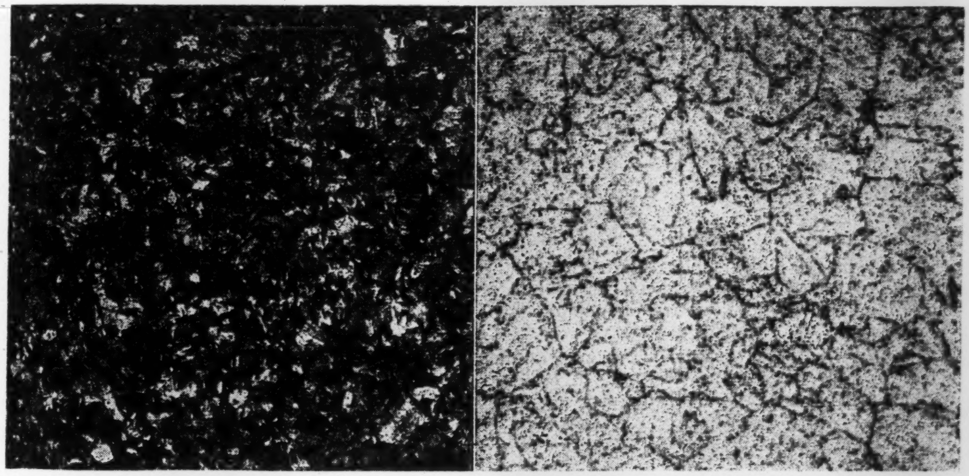
decrease in grain size of one ASTM number increased the average keyhole Charpy impact strength 6 ft-lb. For each of the compositions studied, a grain size of No. 6 or finer, which was associated with a discontinuous grain boundary carbide network, produced keyhole Charpy impact strengths greater than 30 ft-lb even when little or no ferrite was present in the quenched microstructure.

Metallographic studies revealed that



(*a*) Prior austenite grain size finer than No. 8 ASTM rating. (× 100)

(*b*) Carbide precipitate around the delta ferrite, within the grains and some carbide grain boundary network. (× 500).



(*c*) Prior austenite grain size No. 6 ASTM rating. Picric-HCl acid etch (× 100).

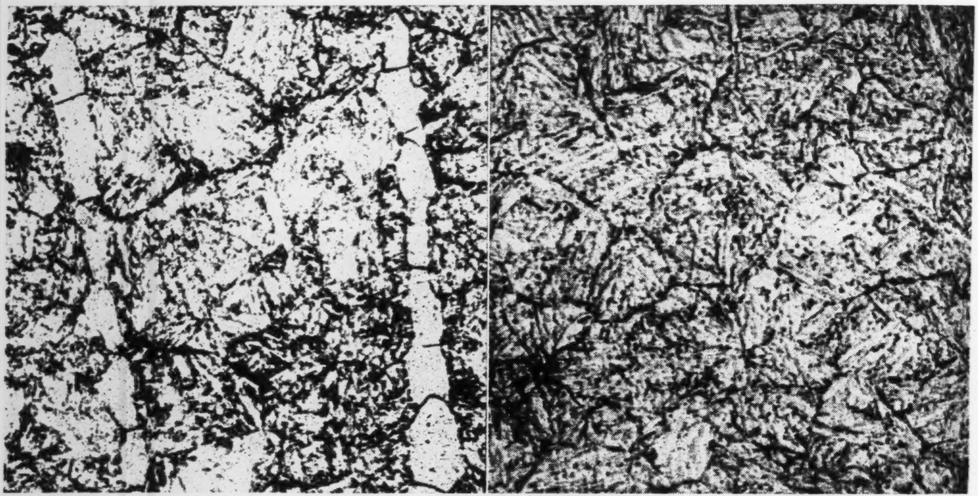
(*d*) Carbide precipitate within the grains and a discontinuous carbide grain boundary network. Electrolytic chromic acid etch (× 500).

FIG. 8.—Comparison of the Microstructure of a Quenched and Tempered 12 per cent Chromium Steel (No. 31) of the Base Composition 0.07 per cent Titanium and 0.04 per cent Aluminum.
(*a*) and (*b*)—Oil quenched from 1650 F, drawn at 985 F.
(*c*) and (*d*)—Oil quenched from 1750, drawn at 1000 F.

some ferrite was present in the 0.92 per cent manganese steel (Code 27), the 0.07 per cent vanadium (Code 30), and the 0.07 per cent titanium (Code 31) steels when austenitized at 1650 F. Ferrite was eliminated from the structure of the vanadium- and titanium-plus-aluminum-treated steels when austenitized at 1750 F but was still present in the manganese steel.

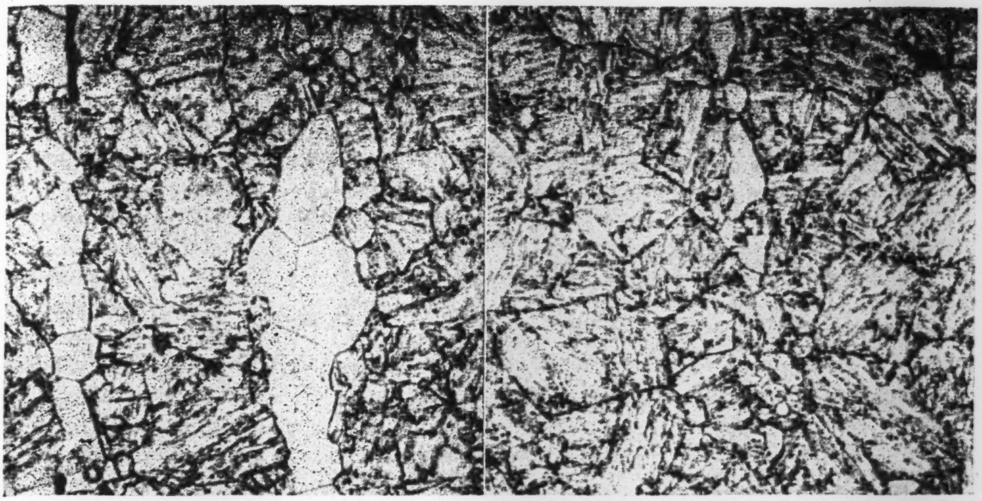
The effect of carbide distribution was such that best impact strength was obtained when the carbides were dispersed randomly rather than along grain boundaries. The addition of carbide or nitride formers such as vanadium, titanium, or aluminum resulted in the presence of undissolved carbides or nitrides after quenching. These undissolved phases acted as nuclei for the carbides that precipitated at the tempering temperature. The result of this process was that the tempered martensite showed a fine precipitate of carbides and nitrides within the grains and less carbide precipitation at the prior austenite grain boundaries. The above description is illustrated by Figs. 5, 6, 7, and 8.

The presence of delta or alpha ferrite in the as-quenched 12 per cent chromium steels also improved impact strength by acting as precipitation sites for the carbides during tempering. The carbides appeared to form preferentially as a ferrite-carbide aggregate around the ferrite areas before precipitating at the prior austenite grain boundaries. An illustration of the improvement in impact strength that can be obtained by controlling microstructure is given in Table V for a commercial heat No. 32, which displayed a wide variation in Izod impact properties. One bar (No. 14) from this heat had an impact value of 37 ft-lb, whereas another bar (No. 6) had an impact strength of 79 ft-lb. These bars were found to have almost identical chemical composition (Table I). However, microscopic examination of the impact specimens machined from the two bars revealed that the structure of the bar with low impact values was essentially tempered martensite with a complete grain boundary network of carbides or carbo-nitrides after tempering. The specimens representing the bar


(*a*) Quenched and tempered structure containing delta ferrite, intergranular carbide precipitate and a discontinuous grain boundary carbide network.

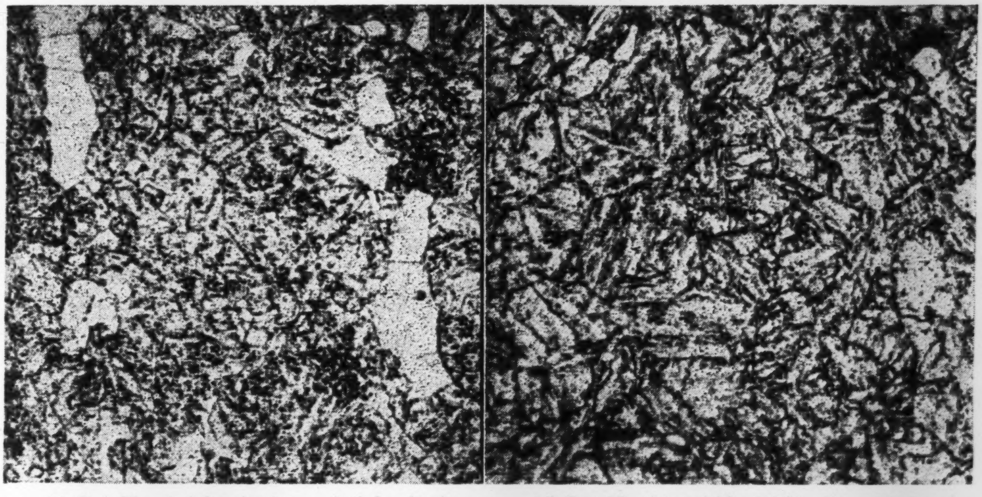
(*b*) Quenched and tempered structure without delta ferrite, with some intergranular carbide precipitate and a semi-continuous grain boundary carbide network.

FIG. 9.—Photomicrographs of Impact Specimens from a Commercial 12 per cent Chromium Steel (No. 32) Representing Bar No. 6 With High (*a*) and Bar No. 14 With Low (*b*) IZOD Impact Values. Austenitized at 1650 F, oil quenched and tempered at 1130 F. Electrolytic chromic acid etch (× 500).



(*a*) Bar No. 6 carbide precipitate around delta ferrite, within the grain and a discontinuous carbide grain boundary network.

(*b*) Bar No. 14 smaller amount of delta ferrite than specimen (*a*), and more carbide grain boundary network.



(*c*) Bar No. 6 delta ferrite and alpha ferrite-carbide precipitate at the prior austenite grain boundaries. Few grain boundary carbides.

(*d*) Bar No. 14 carbides within the grains and a discontinuous carbide grain boundary network.

FIG. 10.—Structure of IZOD Impact Specimens Representing Bar No. 6. High Impact and Bar No. 14 Low Impact Values. Commercial 12 per cent chromium steel (Code 33) after the following heat treatment. Electrolytic chromic acid etch (× 500).

(*a*) and (*b*)—Normalized at 2050 F., Austenitized 1650 F, oil quenched, tempered at 1050 F.

(*c*) and (*d*)—Oil quenched from 2050 F, reaustenitized 1550 F, oil quenched, tempered at 1050 F.

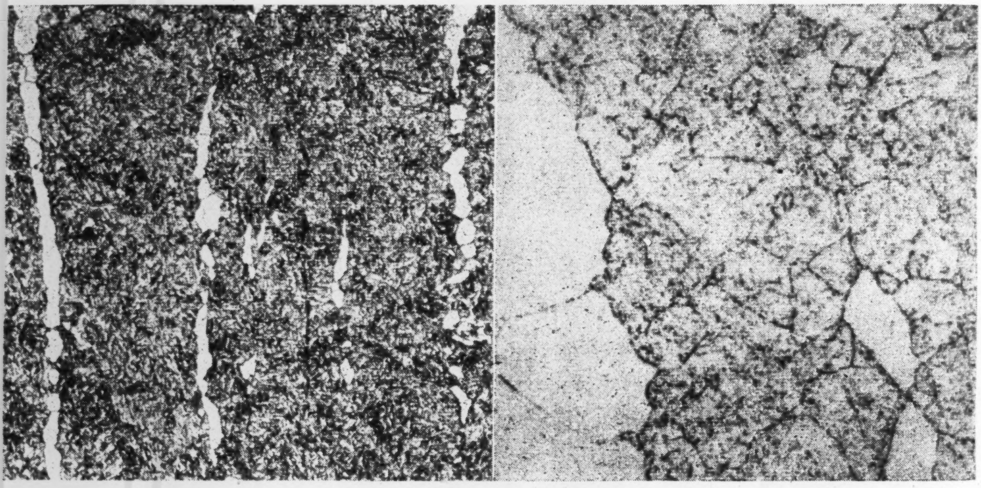
(No. 6) with high impact values contained delta ferrite, with fewer carbides at the prior austenite grain boundaries (Fig. 9).

When bar No. 14 with low impact values was normalized 1 hr at 2050 F, austenitized 1 hr at 1650 F, oil-quenched and tempered at 1050 F to a Brinell

TABLE V.—EFFECT OF HEAT TREATMENT UPON THE MECHANICAL PROPERTIES AND MICROSTRUCTURE OF SEVERAL BARS FROM A COMMERCIAL HEAT OF 12 PER-CENT-CHROMIUM STEEL.

Code	Bar No.	Austenitizing Temperature, deg Fahr[a]	Brinell Hardness after Oil Quenching	Tempering Temperature, Deg Fahr[a]	Brinell Hardness after tempering	Tensile Strength, psi	Yield Strength, psi	Elongation in 2 in., per cent	Reduction of Area, per cent	Izod Impact Results, ft-lb at 80 F	Ferrite Rating, per cent
No. 32....	14	1650	...	1130	241	111 900[b]	82 420	24.5	70.4	(37)[c]	None
					223	106 700	73 430	23.5	71.2	(79)	6–7
	6	2050+	Air cool								
		1650	294	1050	235	...	...	...	...	(36)	5–6
	14	2050	Air cool								
		+1650	344	1050	235	...	...	...	...	(28)	Trace
	6	2050+	311								
		1550	315	1050	235	...	...	...	...	(80)	7–8
	14	2050+	327								
		1550	340	1050	235	...	...	...	...	(50)	Trace
	14	2200+									
		1550	321	1050	255	...	...	...	...	(60)	5–6

[a] Held 1 hr at temperature.
[b] Tempered to 200 to 240 Brinell hardness.
[c] Designates averages.



(*a*) Prior austenite grain size No. 7.5 ASTM rating. Picric-HCl acid etch (× 100).

(*b*) Carbide precipitate around the delta ferrite, within grains and a discontinuous carbide grain boundary network. Electrolytic chromic acid etch (× 500).

FIG. 11.—Photomicrographs of Impact Specimen From a Commercial 12 per cent Chromium Steel (Code 32) Representing Bar No. 14 After Oil Quenching From 2200 F, Reaustenitizing and Oil Quenching from 1550 F + Tempered at 1050 F.

hardness of 235, a small amount of delta ferrite and a fairly continuous intergranular carbide network formed (Fig. 10(*b*)) and the Izod impact value was lowered to 28 ft-lb. When the same treatment was applied to bar No. 6, more delta ferrite and discontinuous intergranular carbide network formed (Fig. 10(*a*)) and the Izod impact strength of the bar was lowered to 36 ft-lb. A similar treatment but with the austenitizing temperature lowered to 1550 F to form some alpha ferrite raised the Izod impact strength to 80 ft-lb for bar No. 6, which contained 7 to 8 per cent of delta and alpha ferrite (Fig. 10(*c*)) and to 50 ft-lb for bar No. 14, which contained little or no ferrite and a discontinuous intergranular carbide network (Fig. 10(*d*)).

The bar (No. 14) which had low impact values and only a trace of delta ferrite was heated to 2200 F for 1 hr to form a small amount of delta ferrite, then austenitized at 1550 F for 1 hr, precipitating a small amount of alpha ferrite, and oil quenched. This bar was tempered at 1050 F to a Brinell hardness of 255 (Table V). The Izod impact strength was increased to 60 ft-lb by this treatment. Photomicrographs of this bar (No. 14) after tempering (Fig. 11) revealed a fine austenite grain size and a more uniform distribution of carbides with fewer grain boundary carbides.

This example clearly illustrates that a microstructure having a fine austenitic grain size together with a random distribution of carbides results in the optimum impact properties. The selection of an austenitizing temperature that results in only a portion of the carbides being dissolved or that results in the presence of as-quenched ferrite will produce this desirable microstructure. Of the two alternatives for improving impact strength, the use of undissolved carbides or nitrides appears to be more desirable than the use of delta or alpha ferrite because of the detrimental effect of ferrite on hot workability and fatigue strength. Since austenite grain size and carbide distribution were closely related, it was not possible to isolate completely the effects of each on impact strength.

The structure obtained by controlling the carbide distribution for improved impact strength should also improve the corrosion resistance by decreasing the amount of intergranular carbides. This influence of carbide distribution upon the corrosion resistance of these steels will be determined in a later program.

SUMMARY

The investigation just described has provided the following conclusions.

Effect of Chemical Composition on Impact Strength:

1. Increasing the carbon content from 0.06 to 0.19 per cent improved the V-notch Charpy impact strength of the 12-per-cent chromium steels. Carbon contents of 0.12 to 0.19 per cent provided impact properties more desirable for many severe commercial applications.
2. Additions of small amounts of nitride- and carbide-forming elements such as aluminum, vanadium, and titanium-plus-aluminum improved the impact strength of 12-per-cent chromium steels tested.
3. Increasing amounts of manganese, nickel, and nitrogen did not appear to improve significantly the impact strength of AISI Types 403 and 410 stainless steel.

Effect of Microstructure on Impact Strength:

1. Within the limits of the compositions and austenitizing temperatures investigated, improved impact strength for 12-per-cent chromium steel was found to be associated with an austenitizing temperature of 1650 F which produced a prior austenite grain size finer than ASTM grain size No. 6 and random carbide distribution after quenching and tempering. It was not possible to separate completely the effects of carbide or carbo-nitride distribution from grain size since they were both related to composition and heat treatment.

2. An average decrease in austenite grain size of one ASTM number increased the keyhole Charpy impact strength 6 ft-lb.

3. A grain size of No. 6 or smaller associated with a discontinuous carbide grain-boundary network after tempering produced a keyhole Charpy impact strength greater than 30 ft-lb at room temperature.

The grain size and the carbide or nitride distribution pattern was varied in the present study by the following procedures: (*a*) The addition of small amounts of vanadium and titanium to form undissolved carbides or carbonitrides at the austenitizing temperatures, (*b*) The addition of small amounts of aluminum which refined the austenite grain size and formed what appeared to be randomly distributed undissolved aluminum nitrides at austenitizing temperatures up to 1750 F, (*c*) Formation of delta or alpha ferrite at the austenitizing temperatures which acted as nucleation sites for the carbide or a ferrite-carbide aggregate to precipitate preferentially during tempering at ferrite grain boundaries before precipitating at the prior austenite grain boundaries, and (*d*) The retention of undissolved chromium carbides at the quenching temperature and refinement of the austenite grain size by the use of low austenitizing temperatures.

Acknowledgment:

The authors wish to express their appreciation to J. J. Heger for his constructive criticism and advice, to E. Van Meter who performed a portion of the testing, and to Mary E. Potter for much of the metallographic preparation and examination.

DISCUSSION

MR. E. T. WESSEL[1]—In general, the effect of increasing carbon in a given steel is to raise the transition temperature, thereby imparting poorer impact properties to the material. However, the authors conclude that, for the 12 per cent chromium steels investigated, increasing carbon in the range of 0.06 to 0.19 per cent improves impact properties and provides a superior material for severe commercial applications. On the basis of the data presented in this investigation, it is difficult to appreciate how such a broad general conclusion can be justified. In the first place, the only series of heats to show an appreciable rise of energy absorption, (at three given test temperatures), as a function of increasing carbon content is the first group (codes 1 to 4) of Fig. 1 and Table II. However, in this series both nitrogen content and grain size decrease as carbon increases. This decrease in grain size and nitrogen content would also tend to produce more favorable impact properties. Is it not possible that these two factors are exerting the predominate controlling influence on properties, while carbon has little or no beneficial effect? In considering the data of Table III, it appears that increasing carbon from 0.12 to 0.19 per cent actually causes a slight lowering of the energy absorption levels as illustrated for the various types of impact tests conducted at 80 F.

Even though increasing carbon is

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[1] Research Engineer, Metallurgy Dept. Westinghouse Electric Corp., Pittsburgh, Pa.

responsible for the reported rise in energy absorption levels at the given temperatures, it is still a precarious conclusion to assume that the impact properties in general have been improved. It is felt that determinations of the entire transition curves (energy absorption *versus* temperature) would be necessary to provide sufficient information for the formulation of this type of conclusion. Observations and subsequent correlations of fracture appearance with the energy absorption curves would also be very beneficial and enlightening. Considering the limited amount of data and the limited temperature range investigated, is it not possible that while the energy absorption levels at the given temperatures may be raised by increasing carbon content, the actual transition temperature has been shifted to higher temperature levels? Naturally the answer to this question is somewhat dependent upon which particular criterion is used to define the transition temperature. This latter aspect itself is the subject of considerable debate.

One further aspect is concerned with the general level of the impact test temperatures used in this investigation. When plotting the energy absorption data of Table II as a function of test temperature, it appears that the authors have been working at the lower end of the transition curves, (temperature range of −40 to 120 F). This indicates that the entire transition temperature curve occurs at an unusually high temperature level for a quenched-and-tempered material. A grain boundary type of fracture may account for this departure from a more normal behavior.

MR. E. J. WHITTENBERGER (*author*).—Because 26-lb laboratory induction furnace heats were used for the majority of the experiments, insufficient samples were available for a thorough evaluation of the influence of composition upon impact transition behavior. It should be noted, however, that in the first bar graph the data for 0 F were beneficially influenced by the higher carbon contents, as were the data obtained at room temperature.

Examination of the 80 F impact data in Table II and Fig. 1 shows a trend toward higher values at higher carbon contents in all series. To be sure this trend is not large in several of the series, but it is always present, regardless of increases or decreases in grain size or nitrogen content. Actually, the variation in nitrogen content was minor and the refinement in grain size may have been associated with the increases in carbon content.

MR. FRANK L. CARR.[2]—The authors are to be congratulated for their extensive work to show the effects of the many variables on the impact properties of these steels.

As a previous speaker has explained, it is uncertain whether or not the difference in impact values are due to a shift in the transition curves. In a recent investigation[3] sponsored by Vacuum Metals Corp., on Iron-Chromium Alloys containing from 0 to 47 per cent chromium and various amounts of nitrogen, oxygen, and carbon, it was found that the carbon and nitrogen had a profound effect upon the transition temperature. Residual oxygen had no measurable effect upon the transition temperature but did have a pronounced effect upon both the hardness and the impact energy level above the transition zone. Although Vacuum Metals Corp. dealt with pure binary alloys rather than steels with thin normal complements of silicon, manganese, etc., it appears that complete charpy

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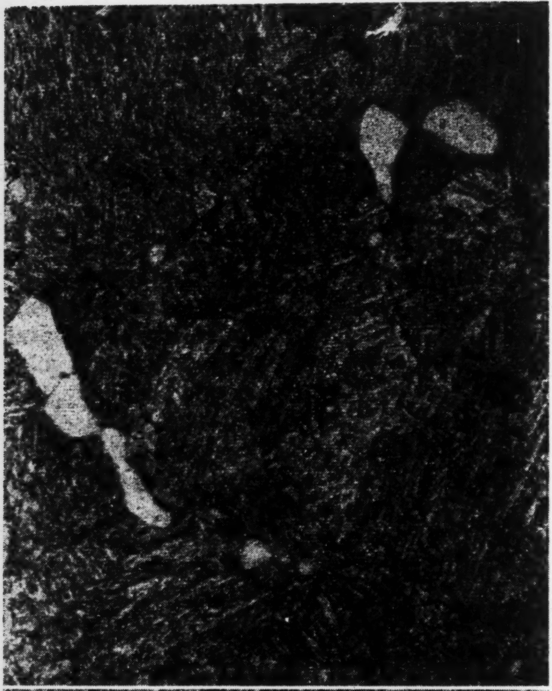
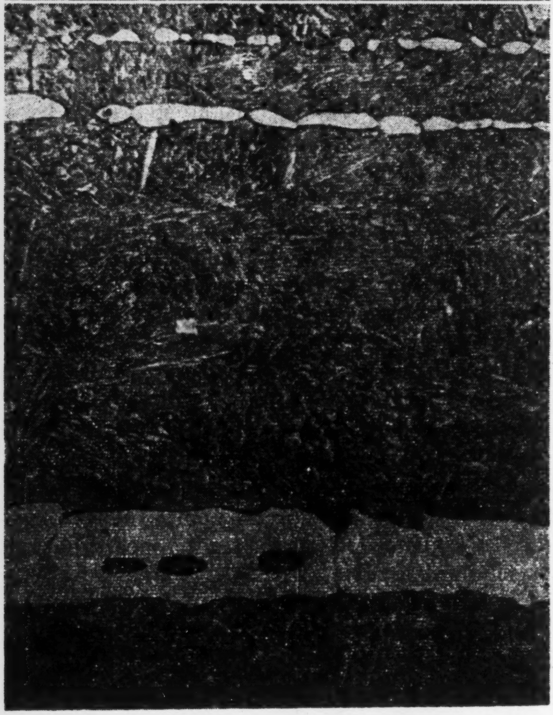
[2] Engineer, National Research Corp., Cambridge, Mass.

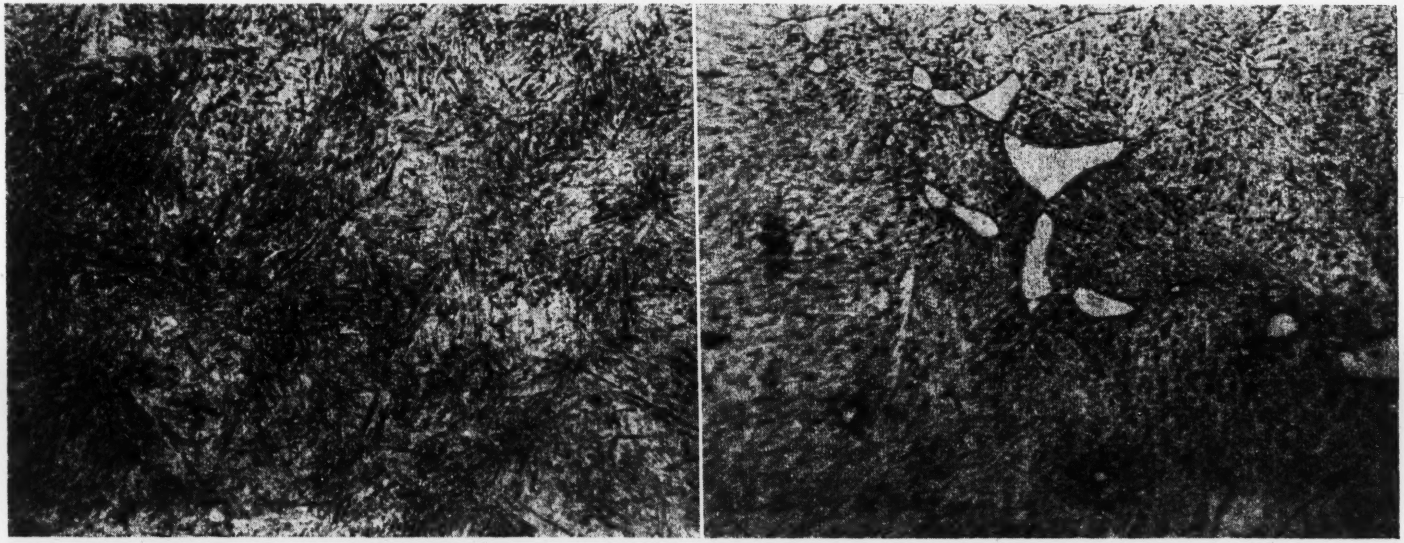
[3] J. Ham and F. L. Carr, "Impact Properties of Vacuum Melted Iron-Chromium Alloys," Vacuum Metallurgy Electrochemical Soc. (1954).



Transverse Section ×300
Rockwell Hardness 20 C
Tempered 120 min. at 1510 F



Transverse Section ×300
As Quenched



Longitudinal Section ×300
Rockwell Hardness 29 C
Tempered 120 min. at 1350 F

Transverse Section ×300
Rockwell Hardness 39 C
Tempered 120 min. at 1150 F

FIG. 12.—Photomicrographs of Modified 403 Stainless Steel.

transition curves would have provided the authors with more definite and detailed information.

MR. WHITTENBERGER.—Oxygen analyses were not obtained, and all of the material available from the small laboratory heats has been used in performing this limited work.

MR. ROBERT G. MATTERS.[4]—The authors have presented an interesting paper on the effect of analysis and structural variations on the impact properties of 12 per cent chromium steels. These steels have been of very considerable interest to us and other people for steam turbine blading applications.

Unfortunately, it is difficult to compare the authors' results with our usual acceptance tests on blading materials, since the standard acceptance test is based upon Izod impact values, rather than Charpy values. The modifications which increase impact strength in the Charpy test should also improve the Izod impact levels.

However, one problem of primary concern is the effect of increased carbon content on the corrosion resistance of these materials. It seems obvious that a substantial increase in carbon content, AISI, type 420, causes a reduction in corrosion resistance at moderate hardness levels compared to the corrosion resistance of type 403. For blading application corrosion resistance is quite important.

MR. WHITTENBERGER.—No data on the influence of composition on corrosion resistance were obtained in the present work. However, the structure obtained by controlling the carbide distribution—that is, to provide a random carbide distribution rather than an intergranular network—should, in addition to enhancing impact strength, improve corrosion resistance.

MR. SAMUEL J. ROSENBERG.[5]—I can not quite go along with the conclusion of the authors as to the effect of carbon content upon the impact properties of these steels. It appears to me from the data shown in the paper that this is a rather doubtful conclusion. There are so many exceptions to this generalized statement that it should be taken with a grain of salt.

Of particular interest was the tremendous—and I mean tremendous—variation that was obtained in the impact properties of the steels. The steels are separated by only a few hundredths of a per cent of carbon. In one case the steel with the highest carbon—around 0.19 per cent—had an impact strength of about 70 ft-lb, as compared with the next lower carbon content, which had about 35 ft-lb. In the ½ per cent nickel steels this trend was just reversed, and this seems to call for a little more study.

There was something else in the paper which was very interesting. The authors give, in tabular form, the effect of decreasing temperature upon the properties of certain of these steels. Some of these tests were duplicated both by keyhole Charpy and V-notch Charpy, and it is very interesting to note that where the values are relatively high, say, around 35 or 40 ft-lb, the impact values ran pretty close together, but as the impact resistance dropped just a little, the keyhole Charpy values were down just a few foot-pounds, but the V-notch Charpy values dropped abruptly, which would indicate that the V-notch Charpy is a much more sensitive criterion of brittle fracture than is the keyhole Charpy test.

MR. WHITTENBERGER.—It is true that the effect of carbon in the 12 per cent chromium series was more marked than in any of the other series, and in particular in the 1 per cent nickel steels the effect of carbon was very much reduced. We have no explanation for this behavior. Possibly, both nickel and manganese additions in some manner masked the effect of the carbon.

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[5] Metallurgist, National Bureau of Standards, Washington, D. C.

Since this paper was written we have made some commercial heats approaching 0.15 per cent maximum carbon content with quite satisfactory impact results. These data were presented verbally to some of the fabricators of these steels. More time will be required to obtain a full evaluation of the merits of the higher carbon contents. With respect to the difference in the V-notch and keyhole Charpy behavior, this, of course, is a very comprehensive and debatable subject, and as suppliers of these steels, we must deal with both the Charpy tests and the Izod test, depending upon the ultimate customer's specification. In this paper we have determined in some instances all three types of impact results, and in others, just one or two of the Charpy results. However, the data in general are much too sparse to draw any extensive conclusions regarding the correlation between, for instance, the keyhole Charpy and V-notch Charpy values.

MR. LEO SCHAPIRO.[6]—In the case of Fig. 11(*b*) of the paper, in which the ferrite band was shown and compared with a good carbide distribution produced by any one of several carbide formers, the comment was made that the free ferrite, or the banded ferrite, contributed to a higher impact value, but that the uniform carbide distribution gave an even better impact value.

As users of this or other materials, we often find it necessary to take cognizance of transverse properties of the materials since loads cannot always be designed to go longitudinally. The word "longitudinally" was never mentioned, but I assume that all the data mentioned had to do with longitudinal properties.

The transverse properties with the banded ferrite would be lower than the one that contained uniform carbides. If the author agrees with this, this is a note of caution for anybody who may think that banded ferrite might be good for impact properties all the time.

MR. WHITTENBERGER.—We certainly concur with Mr. Schapiro. Actually, the last figure was merely used to demonstrate the advantages of the presence of ferrite in increasing the longitudinal impact values. However, the general sense of the paper is that since ferrite is undesirable for other reasons, the achievement of a reasonably fine austenite grain size through the use of the lowest possible austenitizing temperatures together with a random distribution of the carbides which precipitate on tempering appears to be the most desirable structure for achieving optimum impact properties.

MR. W. F. BROWN, JR.[7]—In 1942, G. Bandel published a paper[8] in which he stated that phosphorus appeared, in part, responsible for the 900 F embrittlement of chromium irons. Would the author wish to make any comment on this study?

MR. WHITTENBERGER.—Bandel's results concerning the effect of phosphorus on the 900 F embrittlement of the chromium iron are based on very meager data. In fact, his phosphorus effect is eliminated by a change in heat treatment. Moreover, results of recent studies of the 900 F embrittlement of the chromium irons indicate that the embrittling phase is a chromium-iron compound and the effect of other elements such as phosphorus is minor.

MESSRS. JODEAN MORROW AND G. M. SINCLAIR[9] (*presented in written form*).—The authors mention the detrimental effect of ferrite on fatigue properties of

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[6] Chief Metallurgist, Douglas Aircraft Co., Inc., Materials Lab., Santa Monica, Calif.

[7] Head, Strength of Materials Section, NACA-Lewis Laboratory, Cleveland, Ohio.

[8] G. Bandel and W. Tofaute, "The Brittleness of High Chrome Steels in the Temperature Range of 500 C.," *Arch. fur den Eisenhütten*, Vol. 15 Jan., 1942, p. 307.

[9] Research Associate and Research Associate Professor of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

12 per cent chromium steels. Tests being conducted at the University of Illinois on a modified 403 stainless steel show that there is such detrimental effect on fatigue limit for this material when the hardness is above 20 Rockwell C, but that at a hardness of 20, the fatigue limit agrees very well with that of other ferrous materials.

The chemical composition of the material is 0.28-carbon, 1.15-manganese, 0.016-phosphorus, 0.011-sulfur, 0.48-silicon, 0.12-nickel, 11.12-chromium, 2.84-molybdenum, and 0.29-vanadium. The material was austenitized at 2000 F, marquenched into a 650 to 675 F salt bath, and then tempered at various temperatures between 1510 F and 1150 F to obtain hardnesses ranging between 20 and 40 Rockwell C. In all, nine *S-N* curves were obtained over this hardness range. It was found that the fatigue limit for the low hardness material was in agreement with expected values for ferrous materials of that hardness[10], but at hardnesses above 28 Rockwell C, the fatigue limit was consistently about 15,000 psi lower than expected. Large pools of the microconstituent referred to by the authors as delta ferrite were present as shown in the accompanying photomicrographs Fig. 12 (note similarity to authors' Figs. 9, 10, 11), yet these pools seem to affect the fatigue strength of only the high hardness material. Tentatively, it appears that the strength of the pools and the matrix are approximately the same at low hardnesses while at high hardnesses, the soft pools act as metallurgical notches and govern to some extent the fatigue limit.

MR. WHITTENBERGER.—The detrimental effect of ferrite on the fatigue strength of the 12 per cent chromium steels has been observed in previous United States Steel Corp. studies. These observations prompted the present investigation which was aimed at development of a steel which would have good impact properties without having to resort to a heat treatment or a composition modification that would create ferrite. The authors are indebted to Messers Morrow and Sinclair for their data illustrating the necessity for avoiding ferrite.

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[10] M. F. Garwood, H. H. Zurburg, and M. A. Erickson, "Correlation of Laboratory Tests and Service Performance," *Interpretations of Tests and Correlation with Service*, Am. Soc. for Metals (1950).

AN EMPIRICAL RELATIONSHIP BETWEEN RUPTURE LIFE AND MINIMUM CREEP RATE IN CREEP-RUPTURE TESTS*

By Forest C. Monkman[1] and Nicholas J. Grant[2]

Synopsis

An empirical relationship has been found to exist between rupture life and minimum (secondary) creep rate for a large number of alloys. This relationship should enable one to estimate the rupture life once the minimum creep rate has been determined for a particular creep-rupture test. The effects of such variables as testing stress, temperature, composition, and total ductility were investigated for their effects on the empirical relationship.

Practical considerations usually prohibit thorough long-time testing of materials to be utilized in high-temperature installations designed for as long as ten or twenty years or more. Therefore, there is considerable interest in estimating long-term lives from tests of relatively limited duration, or from tests which can be shortened significantly. While a number of theories of creep and methods of extrapolating short-time data based on these theories have been proposed, none has yet been proven wholly satisfactory. Actually, insufficient testing has been done to effectively check existing extrapolative methods.

The "rate-process theory" was first proposed by Eyring (1)[3] and was first applied to the plastic deformation of metals by Kauzmann (2) and Dushman, *et al.* (3). Subsequently, several methods of extrapolation have been proposed, based on this theory. By assuming that the free-energy barrier in this theory was inversely proportional to the rupture life, Machlin and Nowick (4) derived an equation for rupture life and for creep. Their assumption would require that the rupture life was inversely proportional to the creep rate, which was found to be the case for 2S-O, 3S-O, and pure aluminum by Servi and Grant (5).

Larson and Miller (6) derived an equation relating testing temperature and rupture life on the basis of the "rate process theory." The applicability and some theoretical limitations of their method have been discussed elsewhere (7, 8).

Manson and Haferd (9) and Manson and Brown (10) derived a temperature-rupture life relationship which was a modification to that of Larson and Miller on the basis of experimental data rather than on physical theory. Grant and Bucklin (11) proposed a graphical method for extrapolating and interpolating short-time creep rupture data. At the present time, some long-time-rupture tests in excess of 20,000 and up to 60,000 hr are in progress on alloy S590 which will give a better evaluation of the Grant-Bucklin extrapolation method, and also of the other proposed methods.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

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[2] Associate Professor of Metallurgy, Mass. Inst. of Technology, Cambridge, Mass.

[3] **The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 604.**

TABLE I.—COMPOSITION OF ALLOYS.

Aluminum	Copper, per cent	Iron, per cent	Silicon, per cent	Manganese, per cent	Zinc, per cent	Magnesium, per cent	Aluminum, per cent
Pure	nil	nil	nil	nil	nil	nil	99.995
2S	0.1	0.5	0.1	0.01	0.01	...	balance
3S	0.1	0.4	0.2	1.2	0.01	...	balance
Aluminum-Magnesium	nil	nil	nil	...	nil	up to 5.1	balance
Aluminum-Copper	up to 2.1	nil	nil	...	nil	nil	balance
Aluminum-Zinc	nil	nil	nil	...	up to 20	nil	balance

Titanium	Hydrogen, per cent	Nitrogen, per cent	Oxygen, per cent	Carbon, per cent	Iron, per cent	Titanium, per cent	Others, per cent
75A	0.02	0.15	0.25	0.20	0.25	99.0	0.25
Iodide	0.011	0.006	0.018	0.037	0.013	99.9	

Stainless Steels	Chromium, per cent	Nickel, per cent	Manganese, per cent	Silicon, per cent	Carbon, per cent	Molybdenum, per cent	Vanadium, per cent
Ferritic	up to 13	...	0.85	1	up to 0.2	up to 2	up to 1
Austenitic	up to 20	up to 20	0.1	0.5	up to 0.2	...	...

Nickel and Cobalt Base	Nickel, per cent	Chromium, per cent	Aluminum, per cent	Titanium, per cent	Cobalt, per cent	Iron, per cent	Carbon, per cent	Molybdenum, per cent	Columbium, per cent	Tungsten, per cent
M252	balance	19	1.0	2.5	10	2.0	0.16	9.8	...	...
Nimonic 80	balance	20	1	2	...	0.5	0.05	...	...	...
Nimonic 90	balance	19	1.5	2.5	...	0.5	0.03	...	...	...
S816	20	20	...	...	45	2.5	0.5	4	4	4
S590	20	20	...	...	20	balance	0.5	4	4	4
Inco 700	balance	16	3	2.5	30	4	0.10	2.4	...	...

In the meantime, other data and methods which might further improve extrapolation confidence or point up important physical relationships among the test variables are always welcome and worth close scrutiny. The research described below is one such effort.

EXPERIMENTAL PROCEDURE

Rupture life and minimum creep-rate data were collected from a number of different sources (11 to 28). Rupture life values varied from less than an hour to greater than a year (up to about 20,000 hr). For the majority of tests, the creep rate was determined by using a dial gage which could be read to 0.0002 in., the gage being attached to the specimen holders. A significant portion of the data was obtained from investigations in which extensometers were attached directly on the gage length of the specimen. A few sets of data were from accurate creep measurements. Care was taken to ensure that all data were obtained on equipment meeting the ASTM Recommended Practice 22E[4] for creep-rupture or creep testing.

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[4] Recommended Practice for Conducting Long-Time High-Temperature Tension Tests of Metallic Materials E 22–41), 1955 Book of ASTM Standards, Part 1, p. 1612.

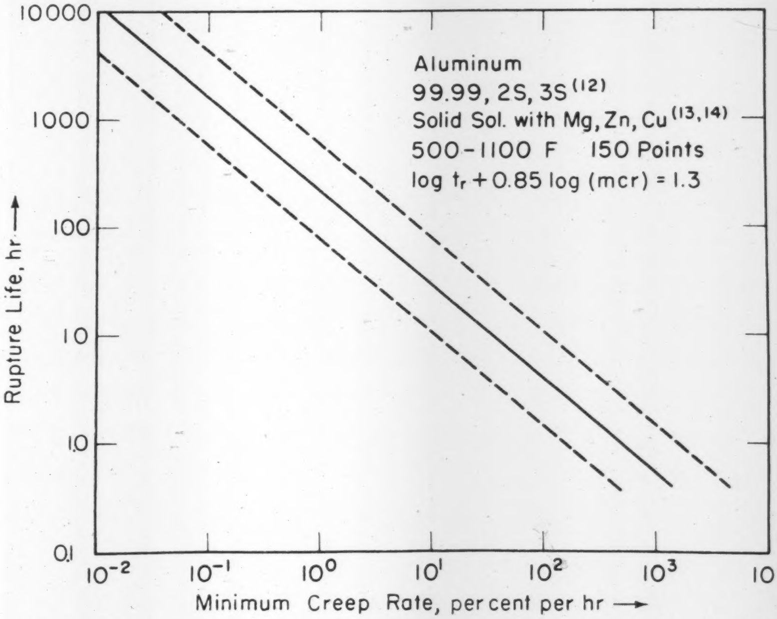

FIG. 1.—Log-Log Plot of Rupture Life *versus* Minimum Creep Rate for Aluminum Alloys.

The numbers in parentheses in these figures refer to the list of references appended to this paper, see p. 604.

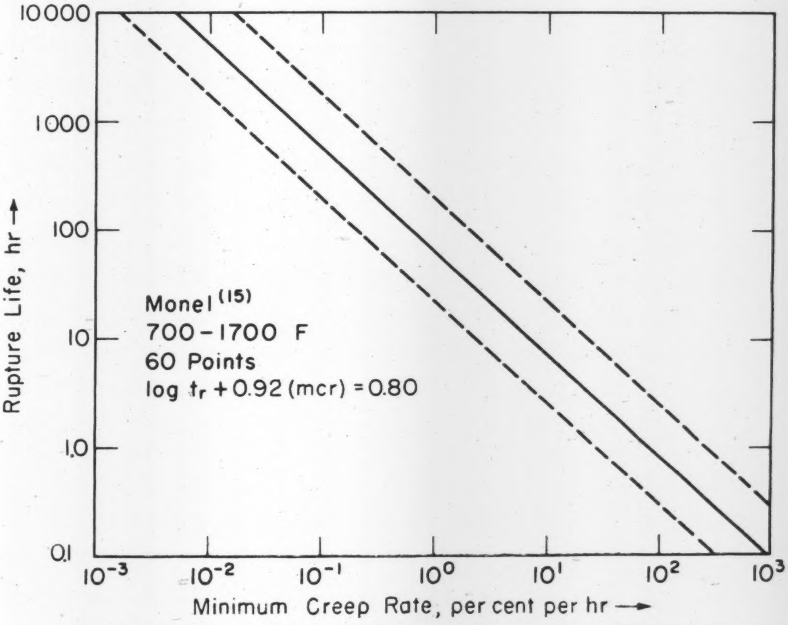

FIG. 2.—Log-Log Plot of Rupture Life *versus* Minimum Creep Rate for Monel.

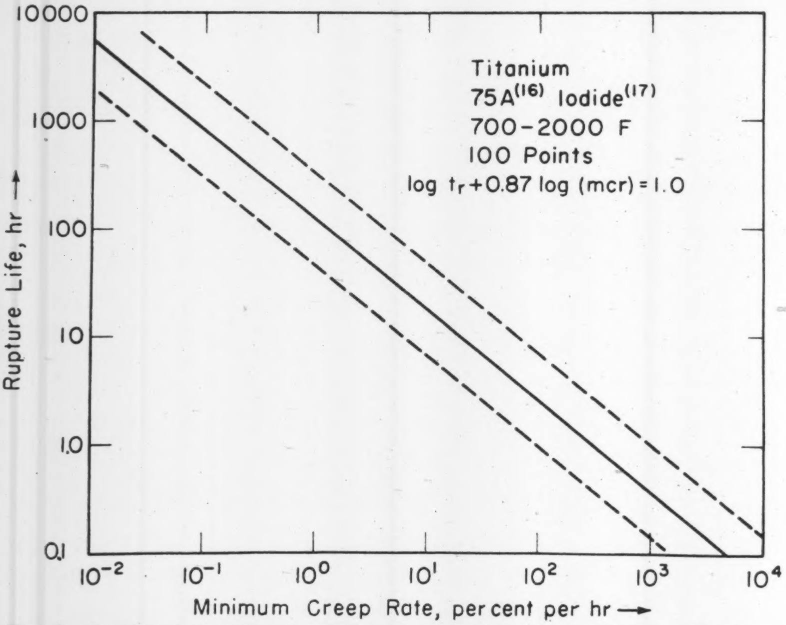

FIG. 3.—Log-Log Plot of Rupture Life *versus* Minimum Creep Rate for Titanium.

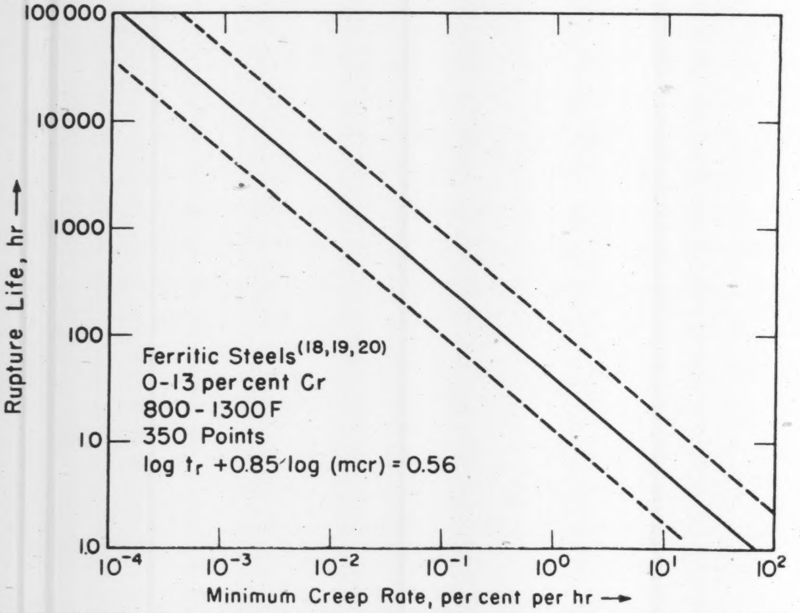

FIG. 4.—Log-Log Plot of Rupture Life *versus* Minimum Creep Rate for Ferritic Steels.

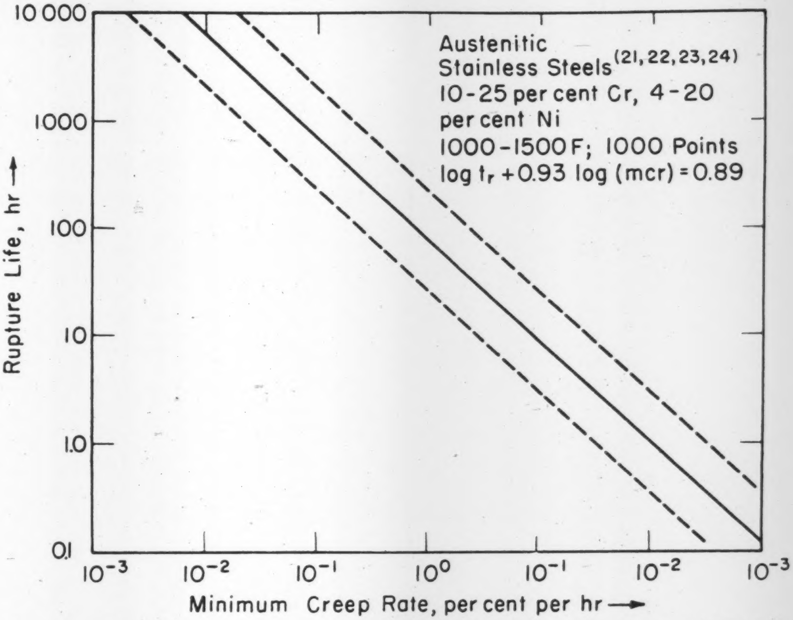

FIG. 5.—Log-Log Plot of Rupture Life *versus* Minimum Creep Rate for Austenitic Stainless Steels.

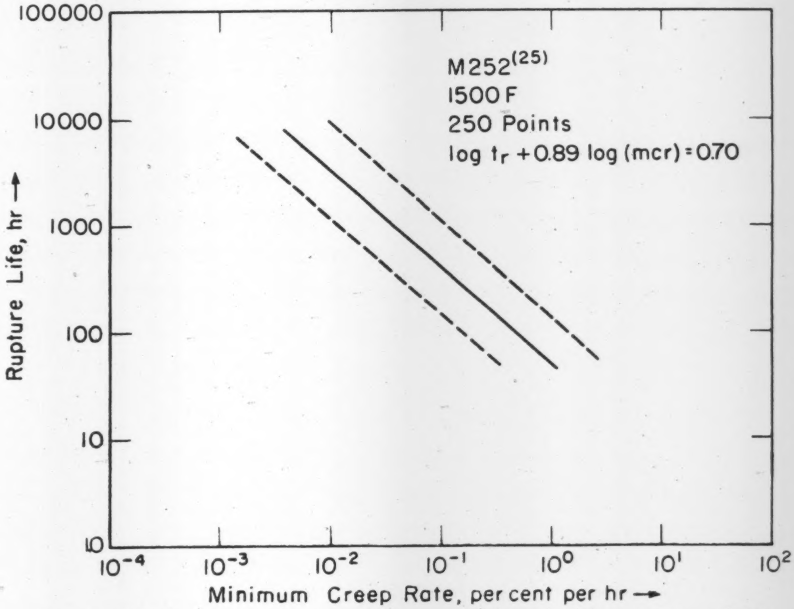

FIG. 6.—Log-Log Plot of Rupture Life *versus* Minimum Creep rate for M252.

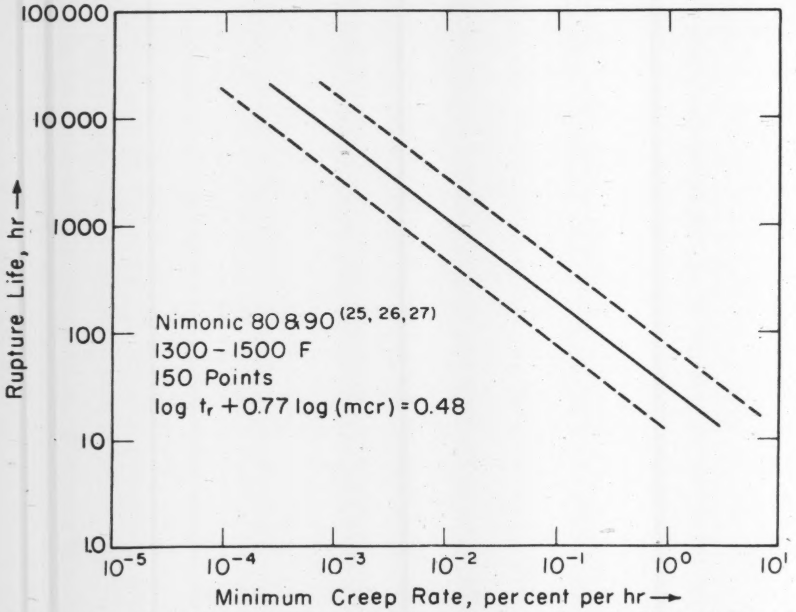

FIG. 7.—Log-Log Plot of Rupture Life *versus* Minimum Creep Rate for Nimonic 80 and 90.

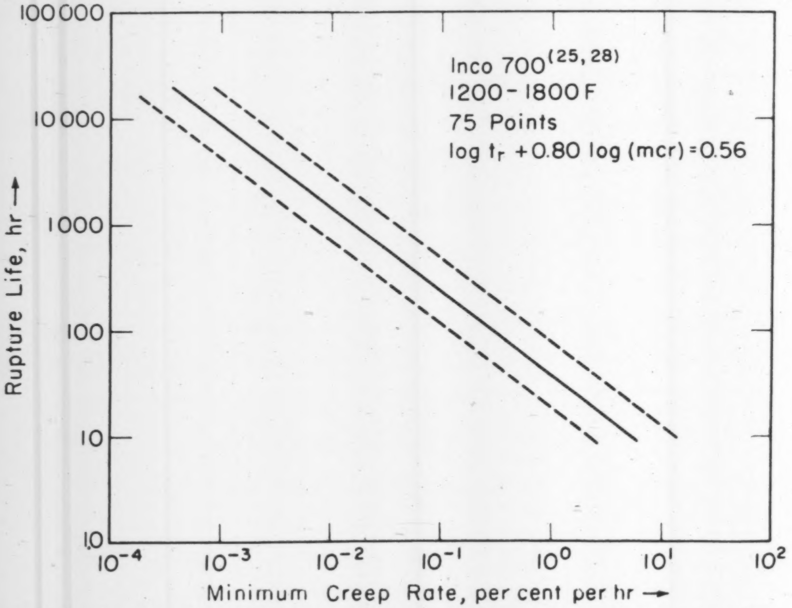

FIG. 8.—Log-Log Plot of Rupture Life *versus* Minimum Creep Rate for Inco 700.

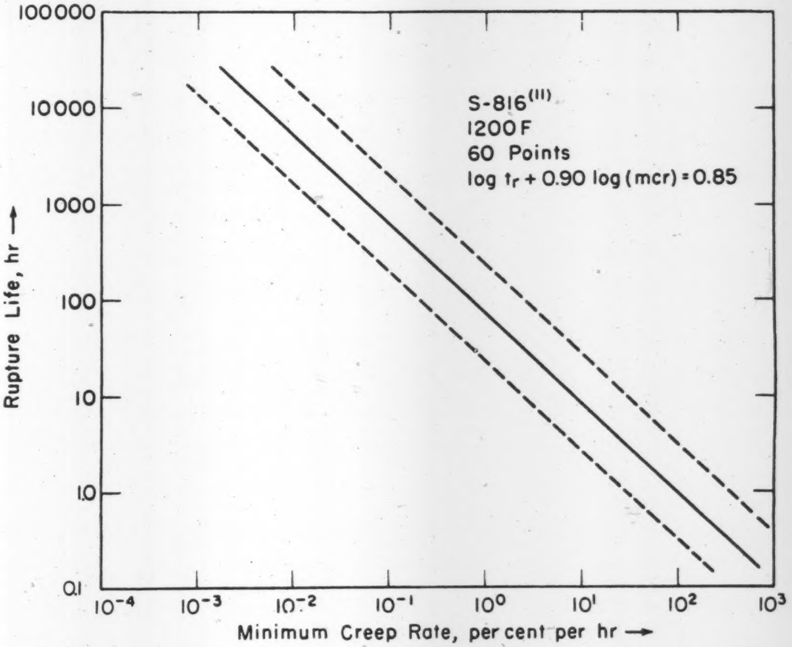

FIG. 9.—Log-Log Plot of Rupture Life *versus* Minimum Creep Rate for S816.

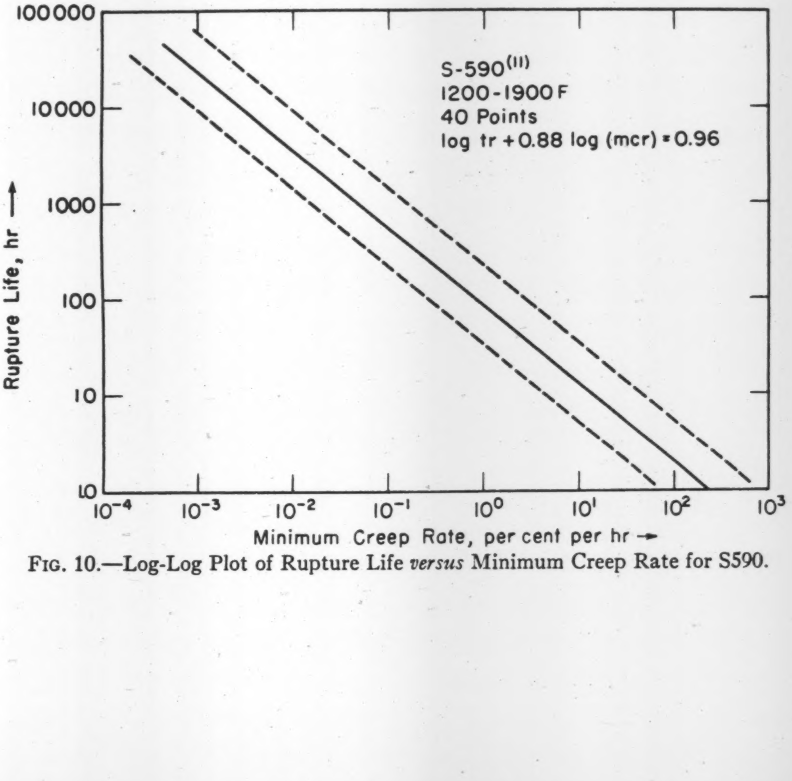

FIG. 10.—Log-Log Plot of Rupture Life *versus* Minimum Creep Rate for S590.

From these data the value of rupture life was plotted against minimum creep rate on log-log paper. The resulting plot for each alloy was carefully analyzed for the effects of testing temperature, final ductility, and composition; in addition, other variables, physical and structural, were examined in less detail.

TEST RESULTS

The compositions of the various alloys are listed in Table I.

The log rupture life *versus* log minimum creep rate plots for the various alloy systems are presented in Figs. 1 to 10. Because of the large number of test values, the individual points have been omitted and only an average curve has been presented; however, a résumé of all the data per curve is listed in Table II. The dotted lines in these plots represent the 2σ limits or a band consisting of 95 per cent of the points. In every case a curve could be selected which was linear within experimental error and could be represented by an equation of the following form:

$$\log t_r + m \log (\text{mcr}) = c \ldots\ldots\ldots (1)$$

where:

t_r = the rupture life,
mcr = the minimum creep rate, and
m and c = constants.

TABLE II.—SUMMARY OF TEST VALUES AND EQUATION CONSTANTS.

Alloy System[a]	Temperature Range, deg Fahr	Number of Tests	Slope, m	Intercept, c	Error Band, Log Cycles of Rupture Life
Aluminum pure, 2S, 3S, (**12**); and Magnesium, Copper, Zinc solid solutions of aluminum (**13,14**)	500 to 1100	150	0.85	1.3	±0.55
Monel (**15**)	700 to 1700	60	0.92	0.80	±0.50
Titanium 75A (**16**): Iodide (**17**)	700 to 2000	100	0.87	1.0	±0.55
Ferritic Steels (**18,19,20**)	800 to 1300	350	0.85	0.56	±0.55
Austenitic Steels (**21,22,23,24**)	1000 to 1500	1000	0.93	0.89	±0.50
M-252 (**25**)	1500	250	0.89	0.70	±0.55
Nimonic 80 and 90 (**25,26,27**)	1300 to 1500	150	0.77	0.48	±0.50
Inco 700 (**25,28**)	1200 to 1800	75	0.80	0.56	±0.55
S-816 (**11**)	1200	60	0.90	0.85	±0.55
S-590 (**11**)	1200 to 1900	40	0.88	0.96	±0.50

[a] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 604.

By analogy to a linear equation of the first order, the constant m corresponds to the slope of the curve and c to its intercept. These constants, the range of testing temperatures, the number of tests per alloy group, and the type of alloy system or systems, and the width of the error band in terms of the rupture life are summarized in Table II.

The error bands in Table II are reasonable for large groupings of stress-rupture data since the reproducibility of rupture life data alone has been reported to have an error band between ±0.27 and ±0.46 cycles of rupture time (29, 30). In addition, some error is introduced into the minimum creep rate data from experimental procedure and the relatively poor precision of measurement and definition.

A slight trend was observed in the data when the individual points were coded with respect to ductility at fracture. For a given creep rate, the specimens showing higher ductility exhibited higher rupture life, that is, such points tended to be near the upper 2σ limit boundary.

Equation 1, it will be noted, is independent of the temperature in the range where the creep rupture tests were made. In some cases, a trend was noted in the data when the individual points were coded with respect to the testing temperatures. However, careful examination revealed that this effect was caused by a change in ductility with testing temperatures, so that actually the relationship may be regarded to be independent of testing temperature.

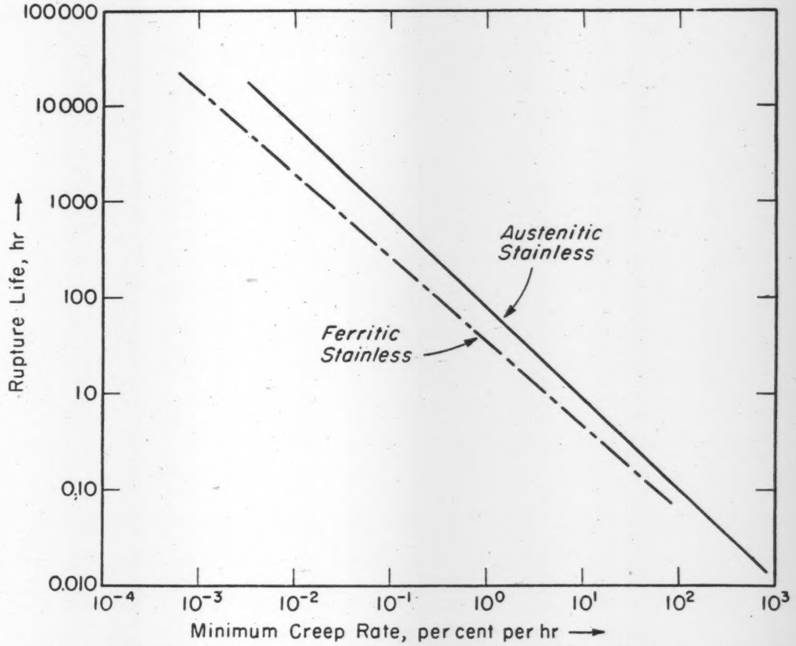

FIG. 11.—Log-Log Plot of Rupture Life *versus* Minimum Creep Rate for Austenitic Stainless and Ferritic Steels.

As seen in Table II, a fairly large chemical variation in a particular alloy group could be adequately represented by a single curve even though in the aluminum alloy grouping, for example, the composition varied from 99.995 per cent aluminum to commercial 3S (5).

Other variables such as aging or heat treatments, oxidation, or recrystallization were not observed to affect the empirical relationship within the limits of the data. Also, the relationship was found to be independent of testing stress.

On the other hand, a significant variation in the constants of the empirical relationship was found among the different alloy groups. Comparison of the curves obtained for the ferritic and austenitic stainless steels provides one possible explanation for such differences. These two alloy systems have approximately the same range of ductility values at fracture but differ mainly in crystal structure. Both sets of curves are shown in Fig. 11. For a given creep rate it can be seen in this figure that the austenitic steels have a greater rupture life than do the ferritic steels. The greater strength of the austenitic type alloys has been reported (20) previously and is attributed to the difference in crystal structure.

The existence of such a relationship as in Eq. 1 between rupture life and minimum creep rate leads to some interesting speculations concerning some

TABLE III.—SUMMARY OF THE CONSTANT IN EQ. 2.

Alloy System	Constant in Eq 2 at a Minimum Creep Rate of	
	10^{-1} per cent per hr	10^{2} per cent per hr
Aluminum	15	38
Monel	5.5	8.0
Titanium	8.0	28
Ferritic Steels	2.6	6.2
Austenitic Stainless Steels	8.0	14
M-252	4.0[a]	8.0[a]
Nimonic 80-90	1.8	8.0[a]
Inco 700	2.2	9.0[a]
S816	5.5	10
S590	6.2	17

[a] By extrapolation.

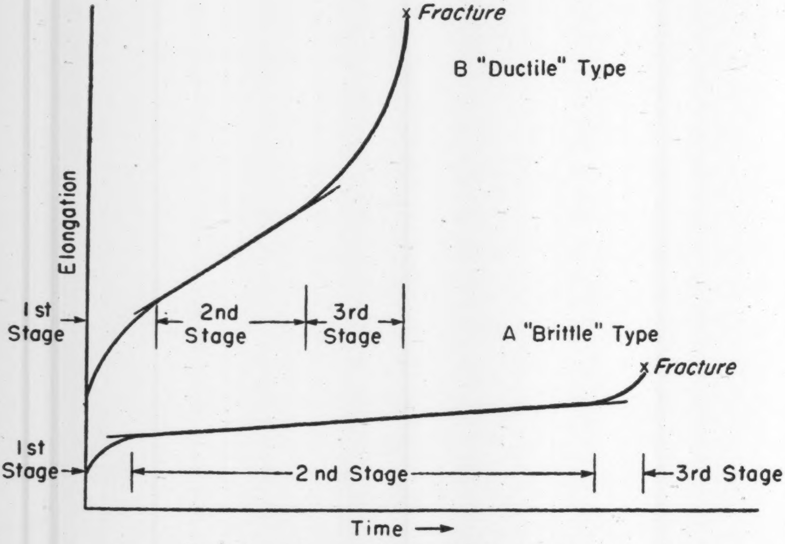

FIG. 12.—Schematic Creep-Rupture Curves.

aspects of the creep process. For a slope m of unity, the empirical relationship can be rewritten in the form:

$$t_r \times \text{mcr} = \text{constant} \ldots\ldots\ldots (2)$$

From Table II it is noted that the slope m was not found to vary appreciably from 1, and therefore Eq 2 is approximately correct for the various alloys.

Dimensionally the constant in this equation is some measure of elongation which is as yet not clearly understood. The values of this constant are listed in Table III for minimum creep rates of 0.1 per cent per hr, which corresponds to a rupture life of about 20 to 150 hr and for 100 per cent per hr, which corresponds to a rupture life of about 0.1 hr. The deformation and fracture characteristics of specimens tested at these two minimum creep rates generally were found to be different. The higher creep rate, 100 per cent per hr, was associated with considerable grain deformation and transcrystalline type fracture, and the lower creep rate, 0.1 per cent per hr, was associated with less deformation and an intercrystalline type fracture. Correspondingly, the values of this elongation constant in Table III are seen to be greater for the higher minimum creep rate, and are reasonably accurate measurements of the ductilities at fracture for these materials in creep-rupture tests.

Nevertheless, one is puzzled, as yet, regarding what this elongation constant signifies. In the case where there is limited ductility, such as in the cast high-carbon cobalt-base alloys, a creep curve is often observed of the type A graphically presented in Fig. 12. Most of the rupture time is occupied by second stage creep and therefore the product of rupture time and minimum creep rate is an approximate measure of the elongation taking place in second stage creep. In this instance, then, the constant in Eq 2 is an approximate measure of second stage elongation.

On the other hand, ductile materials often show a creep curve of type B, in which the minimum creep rate is measured over a relatively short period of time compared to the rupture time. It is also evident that the ratio of second stage elongation to total elongation is quite different for these two types of creep curves (other shapes are not considered for the sake of simplicity).

In spite of these differences, Eq 2 appears to be suitable for a wide range of metals and alloys. It would seem, however, that the elongation values calculated in Table III represent considerably more deformation than can be accounted for by either first or second stage creep. Probably these values are more nearly equivalent to first plus second stage creep, but certainly less than total elongation.

In addition to these speculations is the possibility that the empirical relationship expressed by Eq 1 will permit some short cuts in long-time creep and creep rupture testing and will permit an evaluation of individual test results serving as a guide post rather than as an extrapolation method.

1. By running a number of complete rupture tests, the empirical relationship can be established, permitting an estimate of the rupture life for a selected or desired minimum creep rate and *vice versa.*

2. In long-time tests, once a minimum creep rate has been determined, the rupture life may be estimated from such an empirical relationship which might permit one to forego the necessity of running the test to failure. This is of considerable importance because materials usually run for a long time after reaching the inflection point in the second stage of creep.

3. This empirical relationship enables one to assess the reliability of an individual creep-rupture test as the minimum creep-rate, and rupture life values should fit within a specified scatter band of other tests on the same material to be statistically reliable.

CONCLUSIONS

1. An empirical relationship has been found to exist between rupture life and minimum creep rate having the form:

$$\log t_r + m \log (\text{mcr}) = c$$

Such an equation has been determined for a wide range of alloys based on aluminum, iron, nickel, titanium, cobalt, and copper.

2. Within experimental error, this equation has been found to be independent of testing temperature, stress and chemical variations for a particular alloy system at elevated temperatures. Other factors such as heat treatment, recrystallization, hardness, etc., do not appear to affect this relationship significantly. The variation in the slope m is very small from one alloy system to another.

3. The ductility at fracture and the specific alloy system appear to affect this relationship. A considerable variation in the intercept c was observed from one alloy system to another.

4. The existence of such a relationship for such a wide variety of materials leads to some interesting speculations concerning some aspects of the creep process and points up the need to in-

vestigate the contributions of the various stages of creep to the total amount of deformation in a creep test.

5. This minimum creep rate - rupture life relationship offers promise in estimating long-time creep-rupture data from short-time data and in serving as a check on the reliability of individual creep-rupture data. The relationship is not intended for extrapolative purposes.

Acknowledgment:

The authors wish to acknowledge the constructive discussions and criticisms of the members of the High-Temperature Laboratory at The Massachusetts Institute of Technology, especially those of A. G. Bucklin who helped to activate this investigation. The authors also wish to thank the Bureau of Aeronautics, which sponsored this work.

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DISCUSSION

MR. THOMAS L. ROBERTSHAW[1] (*presented in written form*).—The authors are to be congratulated on their discovery of this useful relationship. On one occasion in the research at Universal-Cyclops Steel Corp. it proved an excellent tool for forecasting rupture life.

This should be an encouragement for the authors in the refinement of their relationship. I have two suggestions for their comment, which are based on a rather meager knowledge of statistical methods.

1. I believe that mathematical means are available for determining the confidence intervals for the slope of the least squares relationship. It would be interesting to see if these values for slope are significantly different, from a statistical viewpoint, from metal to metal.

2. Mathematical means are also available, I believe, for checking the significance of the variance or "error band" differences between the various materials. It appears from the discussion of Table II that much of this scatter can be interpreted as a difficulty in reproducing test results. However, if most materials exhibit similar error bands, regardless of testing temperature, chemical variations, and prior heat treatment, it would seem that a few selective tests on a new material would predict the amount of probable "improvement" by slight variations in these factors.

MR. ERVIN E. UNDERWOOD[2] (*presented in written form*).—The authors have demonstrated the applicability of their Eq 1:

$$\log t_r + m \log (\text{mcr}) = c$$

to a wide variety of creep-rupture data. In conjunction with the theoretical derivation of Eq 1 by E. S. Machlin (1),[3] this expression must be accorded further credence.

In our work at Battelle Memorial

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[1] Universal-Cyclops Steel Corp., Bridgeville, Pa.

[2] Battelle Memorial Institute, Columbus, Ohio.

[3] The boldface numbers in parentheses refer to the list of references appended to this discussion, see p. 620.

Inst., it was preferred to look at expressions of this nature from a more general viewpoint. It seems appropriate at this time to point out explicitly the underlying relationship connecting expressions such as Monkman and Grant's, the Larson-Miller parameters (2), the θ-parameter of Dorn (3), the Z-parameter of Zener and Hollomon (4), and of others (5).

Since all these functions involve a rate of one kind or another, and since the temperature-dependence of the rate of a process is generally expressible by an exponential term, the Arrhenius rate equation is a logical one to consider. It may be expressed as

$$r = Ae^{-Q/RT} \qquad \text{(3)}$$

where r is a rate, A and Q are constants, R is the gas constant, and T is the absolute temperature. If this equation is applied to creep or creep-rupture data, a logical choice of rates would be the (mcr) or $1/t_r$, respectively. Substitution of these rates in Eq 3 yields both forms of the Larson-Miller parameter, P,

$$P_1 = T\,[C_1 - \log(\text{mcr})] \qquad \text{(4)}$$

and

$$P_2 = T\,(C_2 + \log t_r) \qquad \text{(5)}$$

where C_1 and C_2 are constants. This derivation was pointed out by Larson and Miller. Unfortunately, the usefulness of these expressions was diminished by the assumption that the constants should be equal to about 20. In general, the best fit obtainable from the experimental data will demand different values of C_1 and C_2.

Dorn has shown that the Z-parameter, $Z = (\text{mcr})\ e^{Q/RT}$, can be obtained from his θ-function, $\theta = t\ e^{-Q/RT}$. It can also be shown that the Z- and θ-functions are equivalent to Eqs 4 and 5. If, for example, logarithms are taken of the Z-function, we obtain

$$\log Z = \log(\text{mcr}) + Q/2.3RT \qquad \text{(6)}$$

Solving for C_1 in Eq 4, there follows

$$C_1 = \log(\text{mcr}) + P_1/T \qquad \text{(7)}$$

The equivalence of the latter two expressions is apparent, as would be expected since both functions describe the same rate process. This point has been discussed in more detail elsewhere (6, 7).

It can also be appreciated that Monkman and Grant's equation is obtained, at constant temperatures, simply by equating Eqs 4 and 5.

$$T[C_1 - \log(\text{mcr})] = T(C_2 + \log t_r) \qquad \text{(8)}$$

Since C_1 will usually not equal C_2,

$$\log t_r + \log(\text{mcr}) = c \neq 0 \qquad \text{(9)}$$

where c is a constant.

Also, the fact that this expression yields a good straight line can be shown to depend on the behavior of Eqs 4 and 5. If P_1 and P_2 are plotted *versus* log stress, the data can be represented fairly well by straight lines. By equating the straight-line equations at the same value of log stress, and by appropriate rearranging, it follows directly that log t_r and log (mcr) are also related linearly. If, in the log stress *versus* P_1 and P_2 plots, the lines are parallel, Monkman and Grant's Eq 2 is obtained; if the lines are not parallel, Eq 1 results.

MR. J. G. KAUFMAN[4] (*presented in written form*).—The authors are to be congratulated for their very interesting and thought-provoking paper. While some of the facets of this relationship between rupture life and minimum creep rate have been recognized and used for some time by research workers, it remained for Messrs. Monkman and Grant to formally present the relationship and attempt to analyze its significance.

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[4] Research Engineer, Mechanical Testing Div., Alcoa Research Laboratories, The Aluminum Company of America, New Kensington, Pa.

Data obtained at the Alcoa Research Laboratories generally tend to support the existence of a straight-line relationship between log rupture life and log minimum creep rate. In fact, one facet of the relationship was made use of by recognizing a condition which frequently exists with aluminum alloys when the constant *m* in the authors' Eq 1:

$$\log t_r + m \log (\text{mcr}) = c$$

is approximately equal to 1. One way of looking at this special case which the authors did not point out is that when this constant is unity, the log stress *versus* log rupture life and log stress *versus* log minimum creep rate curves are mirror images. An example of this is shown in Fig. 13.

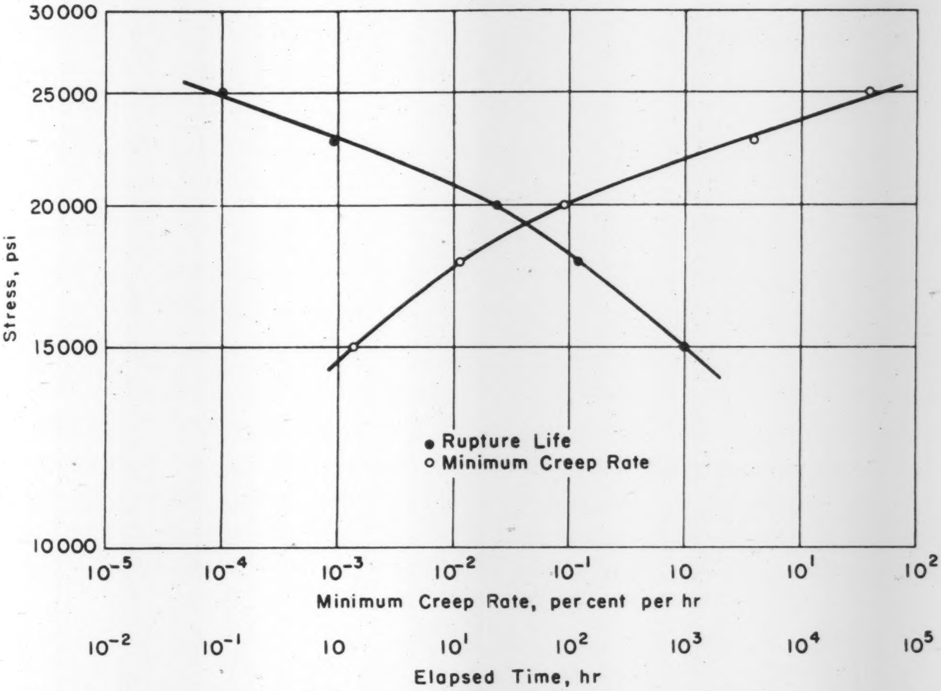

FIG. 13.—Log-Log Plot of Stress *versus* Minimum Creep Rate and Rupture Life for No. 2EC-T6 at 300 F.

Concerning the relationship as a whole, there are several points about which the authors should be cautioned—at least when this relationship is applied to aluminum alloys. Data have been shown by the authors for several commercial non heat-treatable alloys and a few additional non-commercial aluminum alloys, none of which are classified as to temper and all of which have been tested at temperatures from 500 to 1100 F. From these data the authors conclude, without restriction apparently, that the relationship is independent of testing temperature, stress, variations in composition, heat treatment, recrystallization, hardness and "etc.", as the paper says, for a particular alloy system. In making these sweeping conclusions, the authors have ignored a large and very important group of data—that which is obtained between room temperature and 500 F, the range of practical interest for most aluminum alloys. By restricting their range of data for aluminum alloys from 500 F to 1100 F they have excluded all but annealed or greatly softened material. Thus, they have excluded a temperature range in which structural changes occur more slowly with time and which strongly affects the mechanical properties. Had the authors examined data from the lower range, they might not have arrived at such broad conclusions.

In the figures referred to below are presented some of the data obtained in that temperature range at two independent laboratories—Alcoa Research Laboratories and University of California Engineering Research Laboratory (**8, 9, 10, 11, 12**). These data are representative of the very large amount of data available from those laboratories.

Figure 14 shows data obtained at the two laboratories for several lots of 1100 (2S) and 3003 (3S) in annealed and cold-worked tempers (-O, -H12 and -H18) in the temperature range from 90 F to 400 F, a range not shown by the authors. About 150 points are shown. The band shown is that which the authors state will include 95 per cent of the data for 1100 and 3003 regardless of testing temperature. It is true that the data fall into a band, and that if the data obtained on annealed material at all temperatures and the other tempers at the higher temperatures were singled

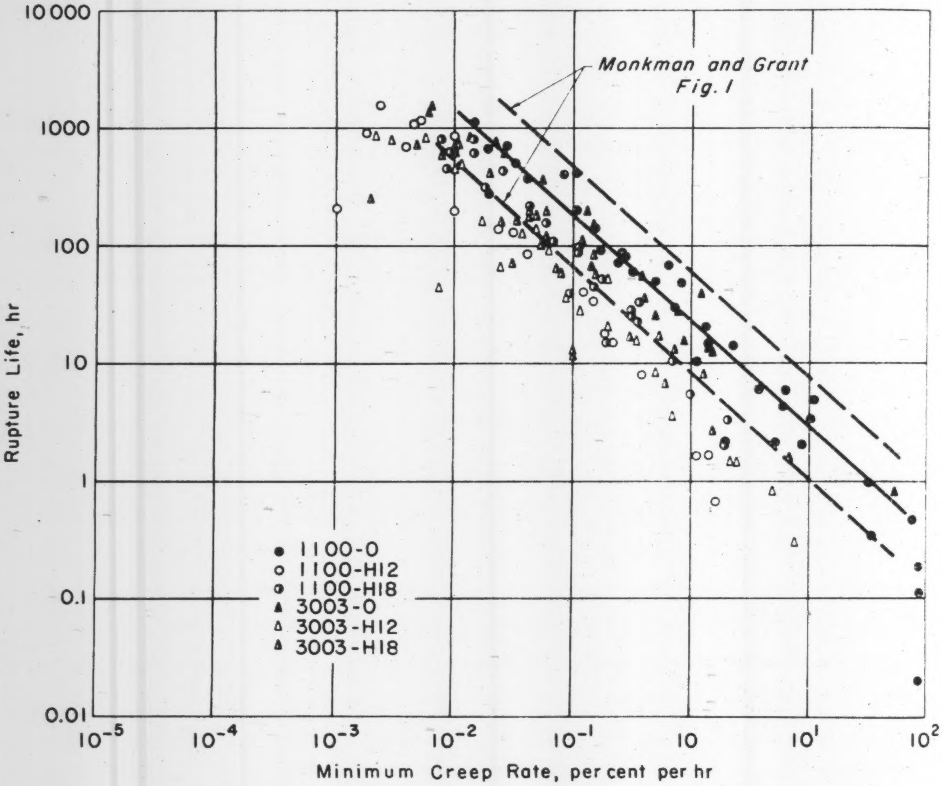

FIG. 14.—Log-Log Plot of Minimum Creep Rate and Rupture Life for 1100 (2S) and 3003 (3S) at 90 F to 400 F.

out, most of it would fall within the authors' limits. Considering only the data for the cold-worked materials, however, all of the points fall below the median and half of them fall below the band. From these data, it is evident that for aluminum alloys, the relationship between rupture life and minimum creep rate differs depending upon the amount of cold work and the temperature.

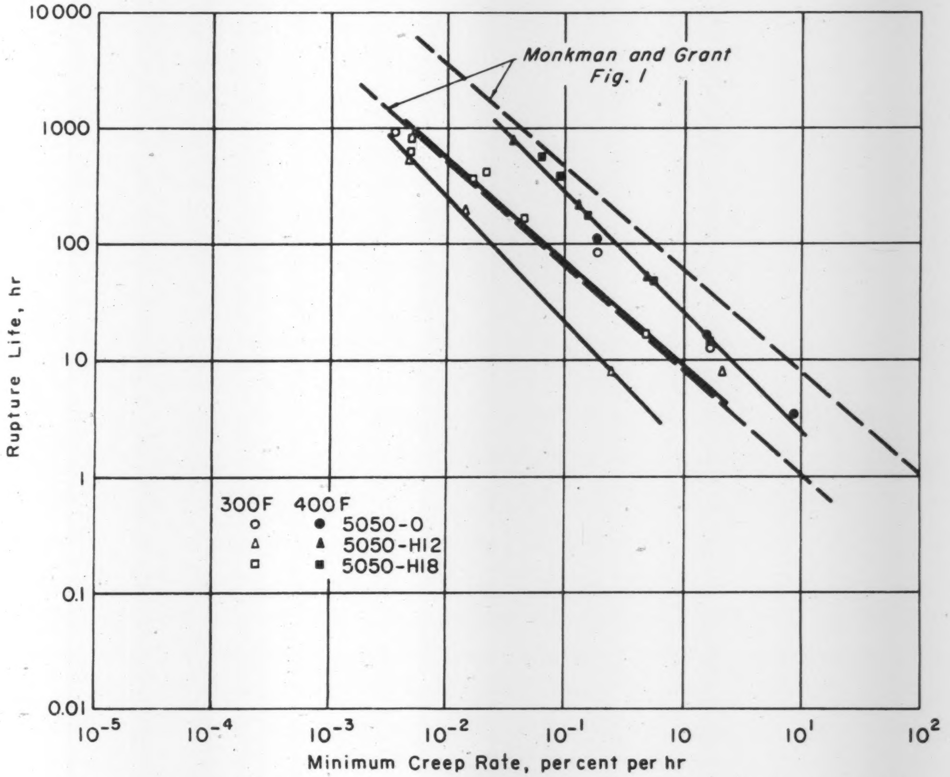

FIG. 15.—Log-Log Plot of Minimum Creep Rate and Rupture Life for 5050 at 300 F and 400 F.

A more detailed representation of the effects of temperature on this relationship may be gained from Fig. 15 which shows data for single lots of 5050-O, -H34 and -H38 at 300 F and 400 F. It should be noted that at 300 F there are three distinct lines for the three tempers; while at 400 F, there is only a single narrow band for the three tempers. Also, this alloy is one which would be included in the authors' band since it is an aluminum—1.2 per cent magnesium binary alloy. At 300 F, only the annealed material fits the band well.

Other examples such as this one could be shown, using heat-treatable alloys as well. Until the temperature is reached at which all of the various tempers behave like annealed materials, a separate line often appears to be present for each temper at each temperature.

The authors also include the statement that this relationship is independent of chemical variations within any particular alloy system and they include high-purity aluminum, non heat-treated alloys, and heat-treated alloys in the same band. It must be assumed that this does not indicate that the authors intend to include all aluminum alloys as one alloy system within this one band, for if we plotted all of our data for commercial aluminum alloys on one chart, the band including 95 per cent of the data would extend more than three cycles on both rupture life and minimum creep rate scales. Data for five aluminum-copper alloys (2014, 2018, 2218, 2618 and 2024) are shown in Fig. 16. Here, the data were obtained at temperatures from 90 F to 700 F. The band of data is rather wide because of (1) differences in composition of the five alloys, (2) differences in temper, (3) differences among various lots of the same alloy and (4) differences in testing temperatures. A band including all of these points would be too wide to be of much practical value.

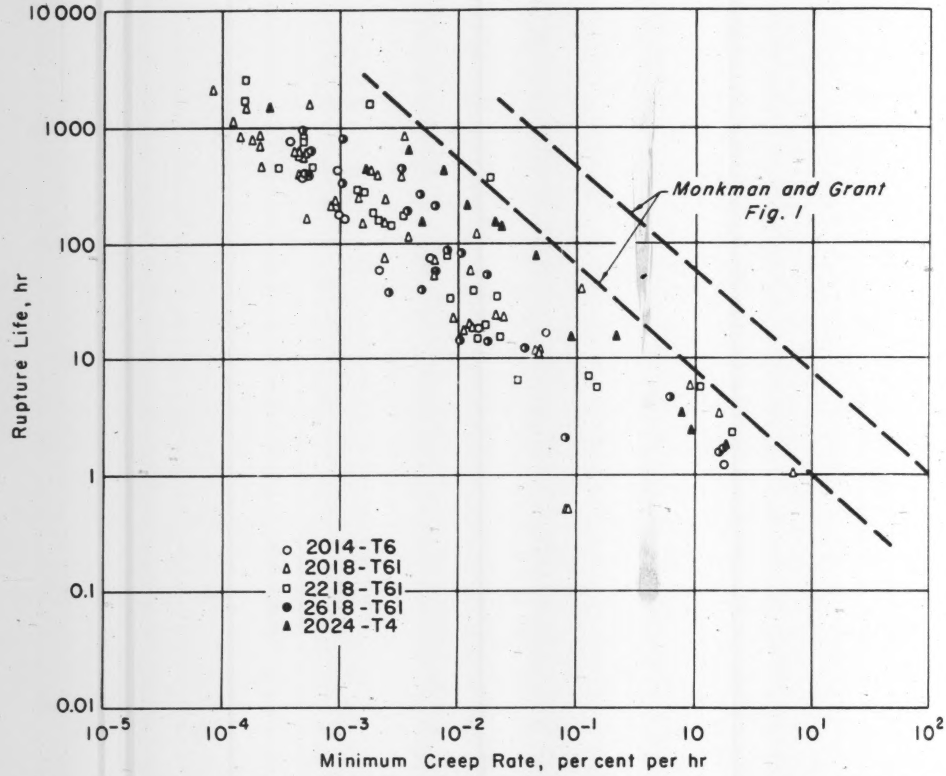

FIG. 16.—Log-Log Plot of Minimum Creep Rate and Rupture Life for 2014, 2018, 2618 and 2024 (Alcoa Research Laboratory and University of California

It should be made clear at this time that the data shown here to exemplify variations resulting from temper, temperature, and chemical composition are only a few examples of the many instances where we can not agree with the authors' conclusions.

This discussion is not intended as criticism. The main point is that what value might be obtained from this relationship will not be enhanced by attempting to throw many aluminum alloys into one single grouping. It is true that a band including data from various alloys and tempers at various temperatures can be established, but it will undoubtedly be too wide to be of much practical value if data below 500 F are included. Furthermore, if bands are set up for aluminum alloys, their widths and the locations of their medians will probably be dependent upon alloy, temper, and testing temperature.

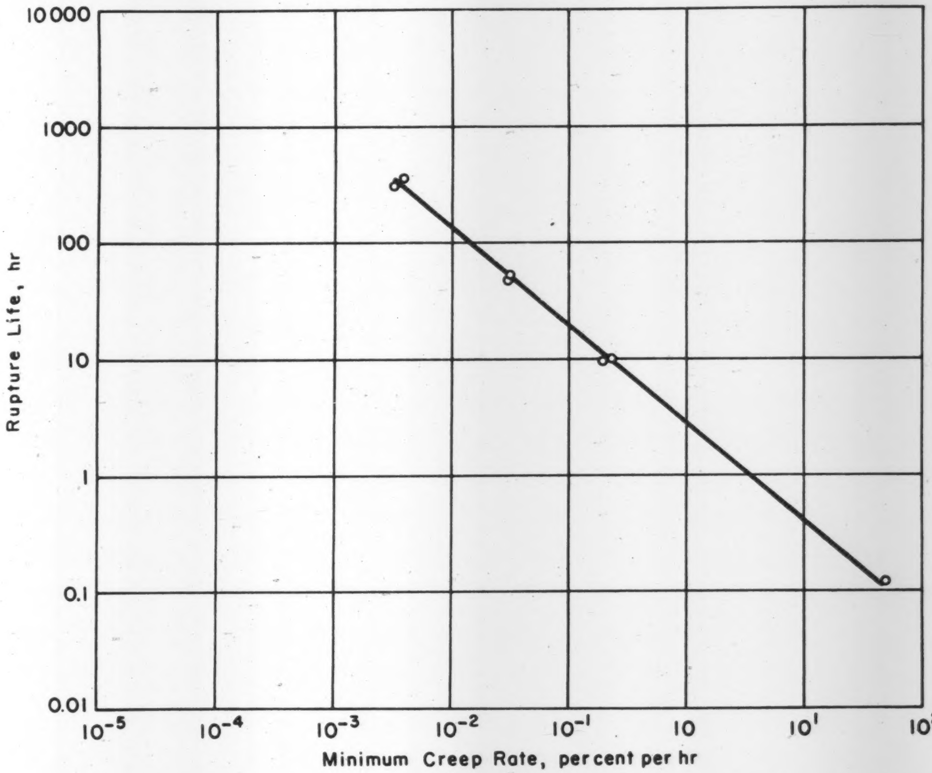

FIG. 17.—Log-Log Plot of Minimum Creep Rate and Rupture Life for Two Lots of X7079-T6 at 300 F.

An example of how this relationship might be useful can be shown. If rupture life and minimum creep rate are plotted for one temper of one alloy tested at one temperature, a single line or narrow band might be obtained. For example, if the data for X7079-T6 at 300 F are plotted, a straight line results as shown in Fig. 17. Such a line would certainly be useful in pointing out erratic data. In addition, this line might be useful, as the authors point out, in estimating either minimum creep rate or rupture life at some stress where one of these factors is known and we wish to estimate a value for the other. For instance, if a minimum creep rate of 0.000001 in. per in. per hr (10^{-4} per cent per hr) has been determined for some stress in a test that has not been run to failure, such a relationship as that shown in Fig. 17 would help in estimating the rupture life for that stress, around 10,000 hr.

Unfortunately, any such extrapolation for rupture life depends on an accurate value of minimum creep rate having been obtained. It is not always possible upon discontinuing a test to be sure that a good value for minimum creep rate has been obtained, since the inflection point, indicating a true minimum slope, may not yet have been reached. To be sure that the inflection point has been reached, it may be necessary to run the test well into the tertiary stage. This is probably the reason why the authors in their conclusions are not too optimistic about using the relationship for extrapolation, and we must agree with that conclusion.

In summary, the authors have put forth a very interesting relationship and our data support the existence of such a relationship. They may have been too optimistic about its limitations as applied to aluminum, at least. It appears that it will be useful in serving as a check on the reliability of creep rupture data. In addition, it may be useful in estimating long-time creep rupture data from short time data for aluminum alloys provided: (1) it is not considered to be independent of too many factors, such as chemical variation, heat treatment, hardness and testing temperature; and (2) that accurate means are available and are used for the determination of the minimum creep rate.

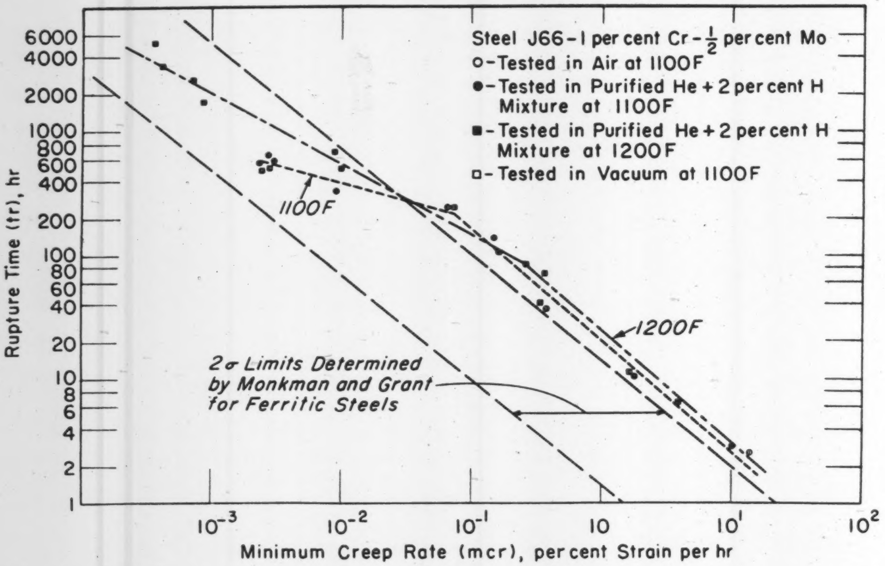

FIG. 18.—Relationship Between Rupture Time (tr) and Minimum Creep Rate (mcr) for a 1 per cent Chromium-$\frac{1}{2}$ per cent Molybdenum Steel Tested in Air, Helium + 2 per Cent Hydrogen or Vacuum at 1100 F and in Helium +2 per cent Hydrogen at 1200 F.

MR. FRANK GAROFALO[5] (*by letter*).—The authors of this paper are to be congratulated for presenting a very interesting analysis. Generally speaking, rupture has been treated in many cases as a phenomenon independent of the plastic flow which precedes it. It is unlikely that this can be shown to be true since rupture is known to be greatly affected by the conditions of plasticity. The present paper gives more evidence of the interrelationship between plastic behavior and rupture.

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[5] Metallurgist, Physical Metallurgy, Edgar C. Bain Laboratory for Fundamental Research, United States Steel Corp., Monroeville, Pa.

For some time we have been investigating the relationship between minimum creep rate (mcr) and rupture time, t_r in ferritic and austenitic steels. The approach has been somewhat different from the approach employed in this paper. In our initial effort an empirical relationship between mcr and t_r was derived from other empirical relationships between initial stress, σ, and mcr and t_r. As discussed recently (13) assuming one or two linear segments for commercial steels in the log σ-log t_r plot an empirical relation between log σ and log t_r for any one temperature may be written

$$\log \sigma = k\sigma \log t_r + k'\sigma \quad \ldots\ldots (10)$$

The slope $k\sigma$ and the intercept $k'\sigma$ are written in this manner since they may depend on the stress range. Similarly, for commercial steels one can write a general relation between log σ and log mcr such as

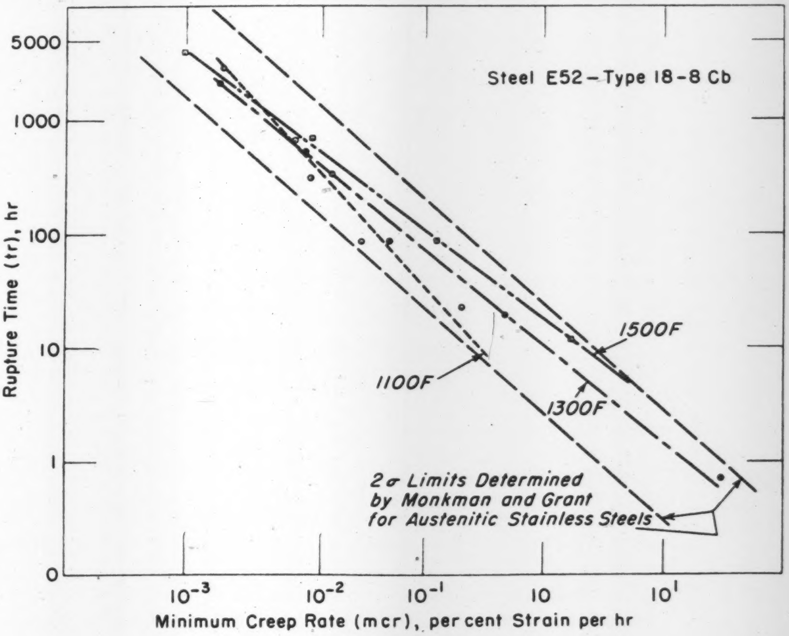

FIG. 19.—Relationship Between Rupture Time (tr) and Minimum Creep Rate (mcr) for an 18-8 Columbium Steel Tested in Air at 1100, 1300 and 1500 F.

$$\log \sigma = K\sigma \log (\text{mcr}) + K'\sigma \quad \ldots (11)$$

By combining Eqs 10 and 11 one obtains

$$\log t_r = \frac{K\sigma}{k\sigma} \log (\text{mcr}) + \frac{K'\sigma - k'\sigma}{k\sigma} \quad ..(12)$$

Equation 12 as should be expected is similar to the one given in this paper.

In our investigation an effort was made to determine the dependence of $\frac{K\sigma}{k\sigma}$ and $\frac{K'\sigma\text{-}k'\sigma}{k\sigma}$ on stress, temperature, and environment. The findings show that for individual steels these quantities do depend in some manner on stress and temperature, and the variations observed do not seem to be related specifically to rupture ductility. Results in air, in a purified mixture of $He + 2$ per cent H, and in vacuum show that under these conditions environment has essentially little effect.

Typical results on one ferritic and one austenitic steel are shown in Figs. 18 and 19 and values of the quantities in Eq 12 are given in Table IV. It should be mentioned that the results obtained in our investigation thus far, for austenitic stainless steels, fall within the 2σ limits established in this paper. As shown in Fig. 18, this is not entirely true for the ferritic steels.

TABLE IV.—SUMMARY OF CONSTANTS IN LOG *tr* – LOG (mcr) RELATION.

Steel	Type	Temperature, deg Fahr	Stress Range, 1000 psi	Slope $\frac{K\sigma}{k\sigma}$	Intercept $\frac{K'\sigma - k'\sigma}{k\sigma}$	Range in Total Elongation, per cent
E 52	18-8 Columbium	1100[a]	37.0 to 60.0	1.09	0.38	15.0 to 40.0
E 52	18-8 Columbium	1300[a]	17.0 to 40.0	0.86	1.0	6.0 to 56.0
E 52	18-8 Columbium	1500[a]	5.0 to 50.0	0.79	1.24	11.0 to 47.0
J 66	1 per cent Chromium-½ per cent Molybdenum	1100[a, b, c]	10.0 to 20.0	0.29	2.0	36.0 to 79.0
J 66	1 per cent Chromium-½ per cent Molybdenum	1100[a, b, c]	20.0 to 35.0	0.91	1.3	40.0 to 59.0
J 66	1 per cent Chromium-½ per cent Molybdenum	1200[c]	4.0 to 10.0	0.57	1.6	50.0 to 113.0
J 66	1 per cent Chromium-½ per cent Molybdenum	1200[c]	10.0 to 15.0	0.90	1.4	78.0 to 109.0

[a] Tested in air.
[b] Tested in vacuum.
[c] Tested in purified helium + 2 per cent hydrogen mixture.

MR. S. S. MANSON[6] and MR. W. F. BROWN, JR.[6] (*by letter*).—The authors have presented an interesting and stimulating study of the possible relationship between creep and stress rupture data. They have proposed a very simple relationship, namely, that the rupture life is inversely proportional to some power of the creep rate. Furthermore, it is stated that this expression is independent of stress, temperature, and many structural factors. However, this relation must be considered as a first approximation since it is derived from a consideration of broad scatter bands on plots of log rupture time *versus* log minimum creep rate. The width of these error bands ranges from ±0.50 to ±0.55 cycles of rupture time. Thus, for a given creep rate, the corresponding rupture time could vary by a factor of 12. The accuracy available using this relation is therefore quite limited.

The existence of such large error bands is attributed to scatter inherent in the data. As supporting evidence for the large scatter, the authors cite the work of Phillips and Sinnott. However, these investigators reported error bands less than $\pm 2\sigma = \pm 0.34$ (total time spread of about 4.8) with the exception of tests conducted at one stress level which scattered by $\pm 2\sigma = \pm 0.46$. The frequency distribution at this stress level was distinctly not normal and was thought to reflect a transition in alloy behavior. Furthermore, the data reported by Phillips and Sinnott cannot be considered as representative of normal stress rupture testing. For example, one-half the data is confined to times less than 2 hr and the test temperature (900 F) was extremely high for their 2SO aluminum alloy. In addition, their practice conformed to ASTM E 21[7] Recommended Practice for short-time tensile testing which permits a ±10 F temperature variation during a test. The ASTM tentative recommended practice E 85[8] for stress rupture testing, limits this variation to ±5 F for temperatures below 1200 F. It would thus appear that the authors' large error band of ±0.50 to ±0.55 time cycles is probably attributable to mixing data from various sources and to neglecting the effects of several primary variables.

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[6] National Advisory Committee for Aeronautics Lewis Flight Propulsion Lab., Cleveland, Ohio.

More quantitative relations between minimum creep rate and rupture time can be derived from time-temperature parameters which have been previously reported. In the authors' references (9) and (10), it was shown that stress rupture data could be successfully correlated by the Manson-Haferd parameter:

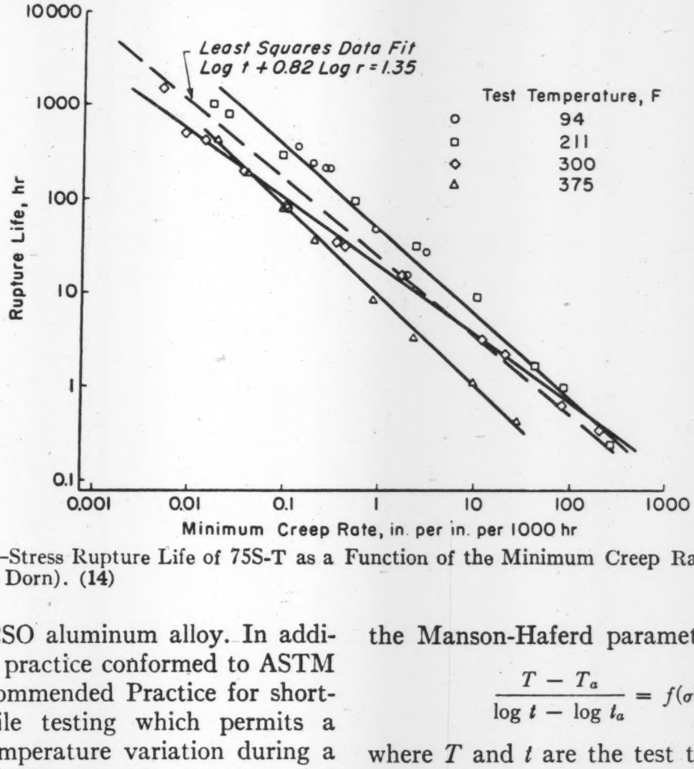

FIG. 20.—Stress Rupture Life of 75S-T as a Function of the Minimum Creep Rate (Flanigan, Tedsen, and Dorn). (14)

$$\frac{T - T_a}{\log t - \log t_a} = f(\sigma) \ldots\ldots (13)$$

where T and t are the test temperature and rupture time, respectively, and T_a and $\log t_a$ are material constants. On the basis of more limited evidence, the following correlating parameter was proposed for creep data:

$$\frac{T - T_a}{\log r + \log r_a} = \emptyset(\sigma) \ldots\ldots (14)$$

where r is the minimum creep rate and r_a is a material constant. The constant T_a appeared to have the same value as in Eq 1.

Dividing Eq 13 by Eq 14, the following

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[7] Recommended Practice for Short-Time Elevated-Temperature Tension Tests of Metallic Materials (E 21 – 43), 1955 Book of ASTM Standards, Vol. 1, p. 1605.

[8] Tentative Recommended Practice for Conducting-Time-for-Rupture-Tension Tests of Metallic Materials (E 85 – 50 T), 1955 Book of ASTM Standards, Vol. 1, p. 1685.

relation is obtained between minimum creep rate, r, and rupture time, t:

$$\frac{\log r + \log r_a}{\log t - \log t_a} = \frac{f(\sigma)}{\phi(\sigma)} = \psi\,(\sigma) \ldots (15)$$

If $\psi\,(\sigma)$ were a constant, this expression would reduce to the same form as proposed by the authors.

It will be of interest to analyze a set of creep and stress rupture data with the purpose of comparing the correlation obtained using the Monkman-Grant relation with that obtained using Eq 15. Unfortunately, published stress rupture data covering a wide range of time and temperature are seldom supplemented by the corresponding minimum creep rate information. Dorn (**14**) has reported rather extensive creep and stress rupture data for the aluminum alloy 75ST. The minimum creep rate for this alloy has been plotted against the rupture time on log-log coordinates in Fig. 20. It will be noted that the scatter band is about one time cycle wide. However, it is clear that this spread is not random but results from a distinct temperature dependence. According to the authors, the equation of the least squares line averaging all the data is the desired correlating relationship. Thus,

$$\log t + 0.82 \log r = 1.35 \ldots\ldots (16)$$

The first step in correlation by means of Eq 15 is to obtain a plot of the Manson-Haferd "master curve" (Eq 13) for the stress rupture data. This may be

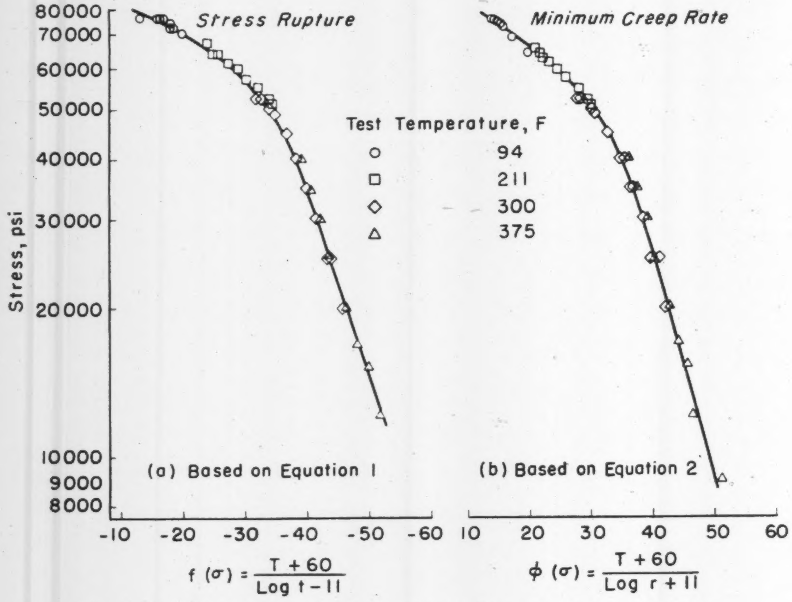

FIG. 21.—Master Curve for 75S-T Stress Rupture and Creep Data.

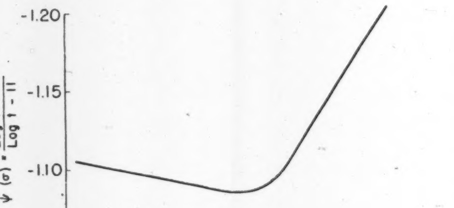

FIG. 22.—Correlating Relation Between Minimum Creep Rate and Rupture Time as a Function of Stress for 75S-T.

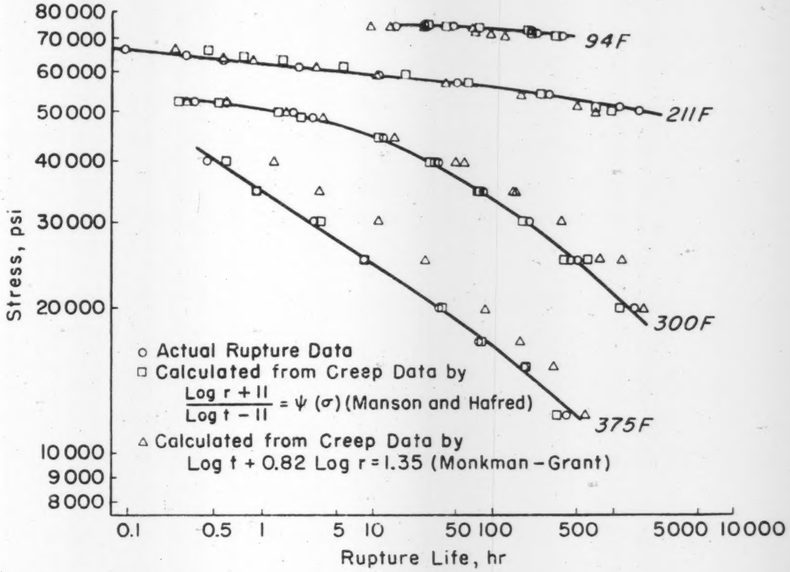

FIG. 23.—Experimentally Determined Stress Rupture Curves and Rupture Times Calculated from Minimum Creep Rate Data for 75S-T.

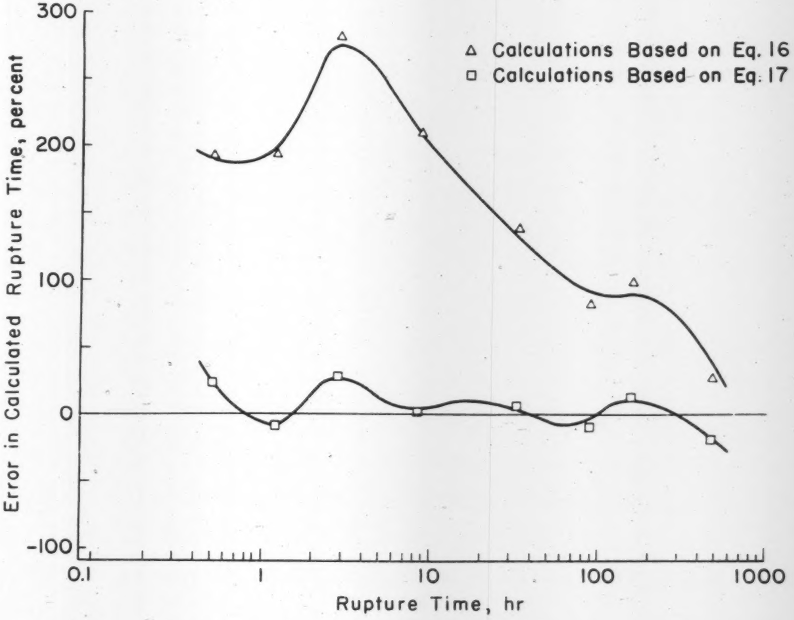

FIG. 24.—Per cent Rupture Time Error at 375 F Resulting from Calculations Based on Eqs 16 and 17 for 75S-T.

done by suitable cross-plotting of the isothermal results as described in the authors ref. 9. The resulting master curve is shown in Fig. 21 (*a*). A similarly obtained master curve for the minimum creep rate data corresponding to Eq 14 is shown in Fig. 21 (*b*). The correlation is then obtained by plotting the ratio between the creep and stress rupture master curves as a function of stress as shown in Fig. 22. The desired correlating relationship according to Eq 15 is:

$$\frac{\log r + 11}{\log t - 11} = \psi(\sigma) \ldots\ldots\ldots (17)$$

where ψ (σ) is a function of stress as plotted in Fig. 22.

A direct comparison of the degree of correlation afforded by Eqs 16 and 17 would be valuable. This may be accomplished by using these relations to calculate the 75ST rupture times from the minimum creep rate data. The results of these calculations are shown in Fig. 23. The rupture strength curves have been drawn through the actual experimental data points. At 94 and 211 F these curves are equally well defined by the points calculated from Eqs 16 and 17. However, at 300 and 375 F, the authors' relation (Eq 16) yields rupture times which are considerably too high, while calculations based on Eq 17 essentially define the experimentally determined rupture curves. The time errors may be better visualized if expressed as a percentage of the values established by the experimental curves. Thus, according to Fig. 24, the time error at 375 F associated with the authors' relation is as high as 275 per cent. In contrast, the errors associated with using Eq 16 do not exceed 30 per cent.

In conclusion, we would suggest caution in accepting the relation between minimum creep rate and rupture time as proposed by the authors. The basic idea that creep information be examined with a view to formulating empirical relationships between flow and fracture is quite sound. However, in attributing the large error bands in the authors' Figs. 1 to 10 to inherent data scatter, the authors discourage development of more quantitative and therefore more useful relations. They also tend to divert theoretical developments by suggesting that the influence of testing and structural variables is negligible.

Messrs. F. C. Monkman and N. J. Grant (*authors' closure*).—We are deeply indebted to Messrs. Robertshaw, Underwood, Kaufman, Garofalo, Manson and Brown for their additions to the data, and for their comments and criticism.

Regarding Mr. Robertshaw's comments, we fully intend to do a more careful mathematical analysis of this type of data but would prefer to use a more carefully controlled test program in which our measurements and the errors in such measurements can also be analyzed. The data, as presented, were subject to all possible errors normally present in creep-rupture testing and therefore did not merit, for the time being, more extensive analysis. That a much narrower scatter band is possible and that relatively few tests can be used for prediction of the relationship is nicely demonstrated in the discussion and contribution made by Mr. Kaufman, as discussed below.

Mr. Underwood's contribution: while we have no reason to deny that a rate process may be operative in creep, the existence of the several modified expressions listed (and this listing should include the expressions of Manson and Haferd and Manson and Brown) emphasizes that the constants in these relationships are all different and therefore yield different results. The derivation or experimental determination of these constants is an important step. Larson and Miller seldom found straight lines for their plots of log stress *versus P*; in fact, the actual shapes of curves in their publication would hardly suggest that a straight-line relationship would be expected for the log stress *versus* log minimum creep rate plot (see also the comments on the discussion of Manson and Brown, below).

We are in complete agreement with the caution expressed by Mr. Kaufman and the data he presented. It is evident that not sufficient care was given to the selection of words in the conclusions, even though certain precautions were taken in our discussion of the data. Specifically, the results and conclusions applied only to the specific materials reported and over the temperature range shown in the captions to the figures and tables. As it turns out, essentially all of our data were in the temperature range where grain boundary deformation processes are important. None of our data or conclusions were intended for extrapolation to low temperature. We certainly should not have suggested that the relationship shown for pure aluminum and its several very simple alloy modifications should apply equally to commercial aluminum alloys since we did point out that important changes in ductility caused a spreading of the scatter band. In this regard, low temperature tests or cold work or certain tempers would bring about the spread so well illustrated by Mr. Kaufman. In fact, in presenting our data, we recognized our inability to combine data from similar commercial alloys in that the curves were deliberately separated for alloys M-252, Nimonic 80 and 90, and Inco 700, even though all of these are nickel-chromium alloys age hardened by means of titanium and aluminum. In this instance, the alloy content variations are not extreme, but ductilities were significantly different and did not permit including all of the data on these alloys on the same plot. Similarly we also separated the similar alloys S-816 and S-590 and the two grades of iron-base alloys, the austenitic and ferritic stainless steels.

We hope that data are available and will be published for relatively low temperatures to determine how the suggested relationship would vary.

In our estimation, the reward of discontinuing a creep test to save many thousands of hours of test time (where this is possible) will stimulate the attainment of more precise secondary creep rate values.

Regarding Mr. Garofalo's contribution, we are indeed pleased to have the confirmation of our austenitic stainless steel plot. Before too great concern can be voiced regarding the disagreement between the two sets of data for the ferritic steels, it should be noted that our data are for the stainless ferritic steels. In line with the observations of Mr. Kaufman (above) and our own separation of commercial alloys, the disagreement may be based purely on a significant difference between types of alloy.

The discussion presented by Manson and Brown deserves careful consideration. The ability of the Manson-Haferd analyses to be fitted to creep-rupture data is not in dispute. The judgment of broader application of their relationship, based on the stress-rupture data of 75 ST, utilizing data at room temperature which we did not utilize in deriving the relationship, falls far short of adequate or fair proof. We would indeed like very much to see the analysis applied to the same data utilized for our presentation.

We cannot apologize for the stress-rupture data used in our analysis for they are a fair cross-section of published data; and contrary to the claim of Manson and Brown, they include constant stress tests as well as constant load, they include data with much more precise temperature measurements than that specified in ASTM Recommended Practice E85 - 50 T, and they include laboratory tests of extremely carefully controlled tests. Unfortunately, when one groups data as we have done, the spread is as we have presented it and cannot be argued away. A few tests will give a narrow band or even a line (see Fig 17, above, of Kaufman), and we have obtained similar results frequently, but that was not the purpose of our presentation.

Contrary to "discouraging development of more quantitative and therefore more useful relations," this work has already been instrumental in stimulating further work in our laboratory and in several others. From a theoretical point of view, we are now examining the role of *ductility* in the creep process, again stimulated by this work.

The existence of the rupture life-minimum creep rate relationship, for the creep-rupture data presented and supplemented by other discussors, does not "suggest that the influence of testing and structured variables is negligible." Instead, it points to the need to examine the creep process in greater detail to see why the relationship is so broadly applicable and how the constants change with changes in composition or structure.

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CORRELATION OF TORSIONAL AND TENSILE PRESTRAIN EFFECTS ON FRACTURE PROPERTIES*[1]

By I. Rozalsky[2]

Synopsis

One of the newer techniques involved in the study of the mechanical behavior of metals is that of prestrain. Subsequent to partially deforming the metal by prestraining under one set of physical conditions—for example, stress state, temperature, strain rate, etc.—the metal thereupon is tested to fracture under a different set of physical conditions.

The object of this investigation was to make a correlation of the effects of room temperature tensile and torsional prestrain upon the tensile fracture properties of annealed copper and spheroidized SAE 1020 steel at various test temperatures from +75 to −321 F. Under these test conditions, the mechanical behavior of the face-centered cubic copper was considered characteristic of that for metals and alloys exhibiting no transition temperature effects. The SAE 1020 steel was chosen as representing those alloys which show transition temperature effects with decreased test temperature, notch effects, etc.

Evidence of rheotropic behavior of the steel was found at lower than transition test temperatures for both tensile and torsional prestrain. The helical type of "wolf-ear" tensile fracture obtained subsequent to critical magnitudes of torsional prestrain was quite pronounced at higher test temperatures but tended to disappear as the test temperature was lowered.

In recent attempts to gain insight into the subject of the mechanical behavior of metals, one of the more popular experimental techniques has been the two-step deformation process. Good summaries are available (**1**, **2**).[3] A metal is subjected to the first deformation or prestrain under one set of physical conditions and then deformed to fracture under another set of physical conditions. Physical conditions may be varied by changing the deformation temperature, stress state, strain rate, etc.

Ripling and Baldwin (**3**) prestrained quenched-and-tempered specimens of SAE 1340 steel in tension at higher than

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] This paper is based upon a portion of a research program conducted in the Metals Research Laboratory, Case Institute of Technology in cooperation with the Office of Naval Research. The data were used as the basis of a thesis submitted to the Case Inst. of Technology by I. Rozalsky in partial fulfillment for the degree of Doctor of Philosophy.

[2] Research Metallurgist, Shell Oil Co., Wood River Research Laboratory, formerly Research Associate, Metals Research Lab., Case Inst. of Technology.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 642.

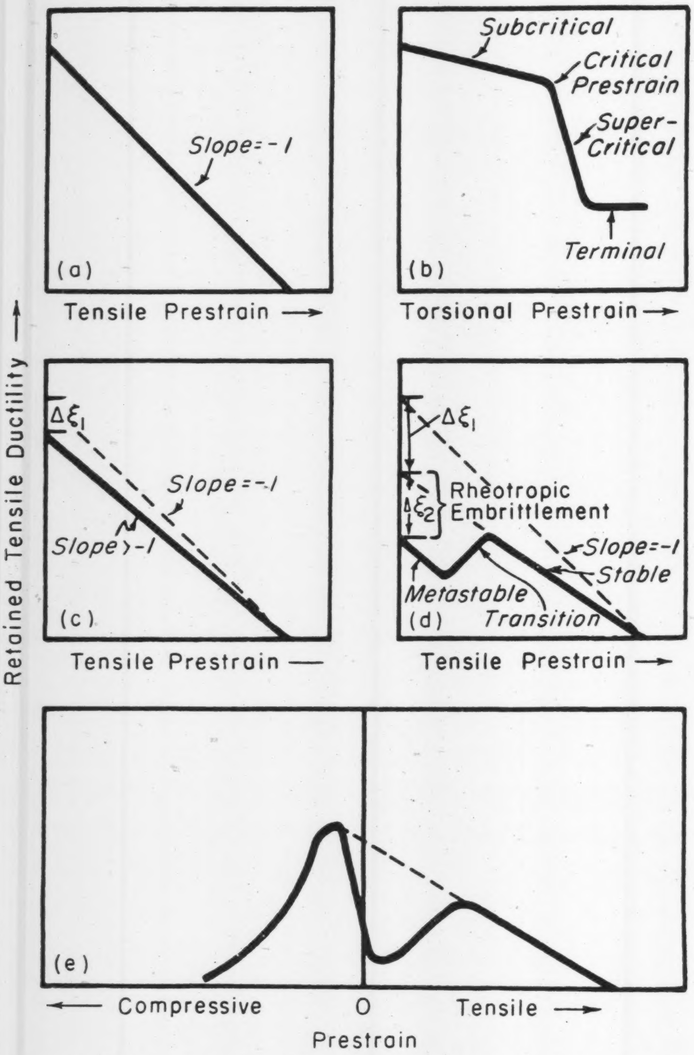

FIG. 1.—Schematic Drawings of the Relationships Between Retained Tensile Ductility and Prestrain (Tensile, Torsional or Compressive).

transition temperatures[4] where the metal was normally ductile and tested these specimens to fracture in tension at lower than transition temperatures where without prestrain the metal would normally exhibit brittle behavior. A plot of the ductility[5] retained at the low temperature *versus* the magnitude of prestrain yielded complex curves. These showed that after an initial decrease in retained ductility for small magnitudes of prestrain, further increases in prestrain increased the retained ductility to a maximum and eventually caused it to pass to zero upon exhaustion of the ductility during prestraining. From their results, Ripling and Baldwin concluded that a part of the ductility deficiency or brittleness at lower than transition test temperatures was strain curable or "rheotropic."

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[4] The transition temperature for a metal is that usually narrow temperature range within which the mechanical behavior (for example, ductility) changes from a ductile to a brittle nature. Apparently, only non-face centered cubic metals exhibit transition temperatures.

[5] The criterion of ductility used in this paper was: $\epsilon = \ln \frac{\text{Original Area}}{\text{Final Area}}$ where ϵ is defined as the maximum natural strain.

Swift (4) prestrained specimens of mild steel in torsion at room temperature and then tested these specimens to fracture in tension at room temperature. With increasing torsional prestrain he observed no appreciable change in the retained tensile ductility until a specimen had been torsionally prestrained to a critical magnitude. Thereupon, with increasing torsional prestrain, there was a rapid continuous decrease in ductility for a subsequent tension test. He also observed a change in fracture appearance from a ductile cup-cone type at less than critical magnitudes of prestrain to a brittle, helical "wolf-ear" type at greater than critical magnitudes of torsional prestrain.

Figure 1(*a*) to (*e*) indicates schematically the tensile deformation behavior for metals subsequent to various types of prestrain. The ordinate axis indicates the retained tensile ductility, whereas the abscissa axis indicates the magnitude of prestrain. Terminal points of the curve on the abscissa axis or the ordinate axis indicate single deformation processes where the ductility is exhausted exclusively under either prestraining or testing conditions, respectively.

Figure 1(*a*) shows the deformation behavior for simple tensile prestrain when the prestrain temperature and test temperature are identical. This type of curve is found for face-centered cubic metals which exhibit no transition temperature or, at higher than transition test temperatures, for metals which do exhibit transition temperature behavior. The curve is a straight line of 45-deg slope.

Figure 1(*b*) shows the deformation behavior for torsional prestrain under the conditions stated above for Fig. 1(*a*). Here the relationship is not as simple as that given above. One should realize, however, that the strain states for tensile and torsional prestrain differ from each other as do their respective stress states. This type of curve has been obtained in several investigations for a number of ductile, relatively pure metals and single phase alloys (5, 6).

Figure 1(*c*) shows for metals exhibiting a transition temperature the effect of prestraining in tension at a higher than transition temperature, T_1, upon the retained tensile ductility at another higher than transition temperature, T_2, where $T_1 > T_2$. Here $\Delta\epsilon_1$ indicates the difference between the ductilities of the metal at the given temperatures. This type of behavior was obtained for specimens of quenched-and-tempered (above 800 F) SAE 1340 steel prestrained in tension at room temperature and then tested in tension at −321 F (3). A similar type of curve was obtained for 24 S-T 4 aluminum (no transition temperature) by prestraining at room temperature and testing at −321 F (7).

No analogous investigation has as yet been conducted to determine the deformation behavior for torsional prestrain at different higher than transition prestrain and test temperatures when the prestrain temperature is higher than the testing temperature.

Prestraining in tension at a higher than transition temperature and testing in tension at a lower than transition temperature yields the relatively complex curve illustrated in Fig. 1(*d*). Upon extrapolating the stable portion of the curve to the ordinate axis, Ripling and Baldwin (3) concluded that the low-temperature ductility deficiency $(\Delta\epsilon_1 + \Delta\epsilon_2)$ of quenched-and-tempered SAE 1340 steel was in part $(\Delta\epsilon_2)$ rheotropic, that is, strain curable.

Ripling and Baldwin (8) found that prestraining SAE 1340 steel specimens in compression at higher than transition temperatures was also capable of restoring part of the tensile ductility normally denied the steel at lower than transition test temperatures, in fact, at much lower magnitudes of prestrain than by prestrain in tension (see Fig. 1(*e*)). The effect of torsional prestrain under similar conditions has also not been determined.

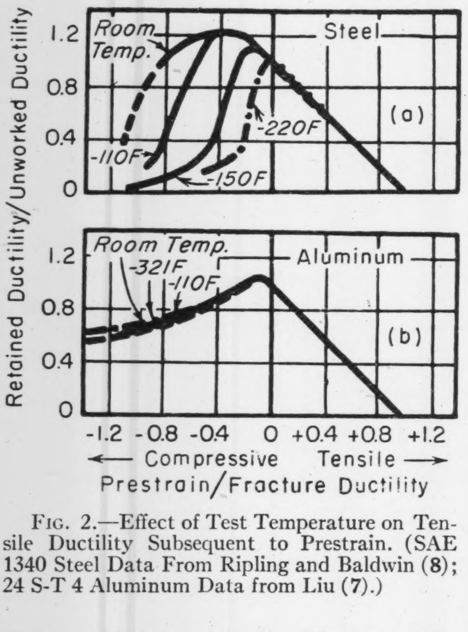

FIG. 2.—Effect of Test Temperature on Tensile Ductility Subsequent to Prestrain. (SAE 1340 Steel Data From Ripling and Baldwin (8); 24 S-T 4 Aluminum Data from Liu (7).)

Figure 2(*a*) previously presented by Ripling and Baldwin (8) shows that after an initial increase in the retained ductility for small compressive prestrains (rheotropic recovery), there is a rapid ductility decrease with increased magnitudes of prestrain. Was this ductility drop-off due to "microcracks," the proposal made by Backofen, *et al.* (5, 6) for the ductility decrease for torsional prestrain? The magnitude of compressive prestrain at which the rapid ductility drop-off takes place recedes to smaller and smaller values with decreasing temperatures (see Fig. 2(*a*)). Would the ductility drop-off obtained by Swift (4) subsequent to torsional prestrain similarly recede under analogous conditions?

But the precompression by Liu (7) of 24 S-T 4 aluminum, a ductile face-centered cubic metal, showed (see Fig. 2(*b*)) no ductility drop-off as severe as that for steel. Does this mean 24 S-T 4 aluminum is insensitive to microcracks? On the other hand, copper (also a ductile face-centered cubic metal) showed a ductility drop-off after pre-torsion at a magnitude of prestrain where it was claimed cracks did occur (5).

The objective of the present investigation was a correlation of the effects of tensile and torsional prestrain upon the tensile fracture properties (retained ductility and fracture stress) for two metals at various testing temperatures. The metals chosen were copper, crystallizing in the face-centered cubic system and, concomitantly, showing no transition temperature, and spheroidized SAE 1020 steel, which does have a transition temperature associated with its mechanical behavior.

MATERIAL AND PROCEDURE

The materials used in this investigation were (*a*) annealed electrolytic tough pitch copper and (*b*) plain carbon SAE 1020 steel in the spheroidized condition.[6]

Torsional prestrain specimens of the type shown in Fig. 3(*b*) were twisted various amounts at a strain rate of approximately 120 deg per min. The plastic shear strain, γ, at the surface of the reduced section of a twisted torsion specimen was calculated from the relation:

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[6] Details of the heat treating, prestraining and testing procedures used in this investigation are found in the Appendix.

$$\gamma = \frac{R\theta}{L}$$

where:

γ = plastic surface shear strain (circular bar),
R = radius of bar,
θ = total twist in radians, and
L = length of bar over which twisting occurs.

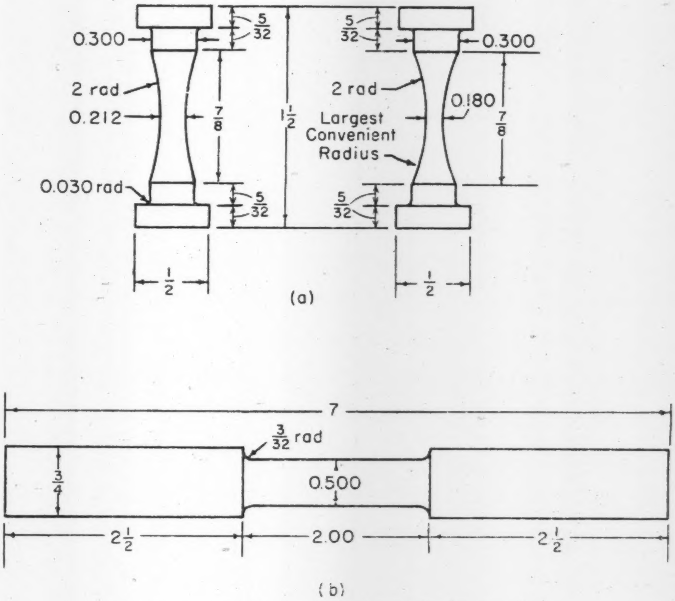

FIG. 3.—Prestrain and Tension Test Specimens.
(*a*) Tensile Prestrain or Test Specimens.
(*b*) Torsional Prestrain Specimen.

Although some doubt has been expressed concerning the validity of the above expression for large magnitudes of strain (**9**), the author considered the use of the above simple equation adequate for this investigation.

After the desired magnitude of torsional prestrain had been effected, a tension test specimen of the type shown in Fig. 3(*a*) was machined from the center of the reduced section of the torsion specimen. Because of the linear strain gradient with respect to the radius of the twisted bar ($\gamma = 0$ at the center), the magnitude of shear strain at the surface of the reduced section of the tension test specimen was readily calculated.

Tensile prestrain was obtained on specimens of the type shown in Fig. 3(*a*). It was not considered necessary to remachine specimens after tensile prestrain. All tensile prestrains and tension tests were made on a hydraulic tension testing machine with a crosshead speed of 0.05 in. per min.

RESULTS AND DISCUSSION

Retained Tensile Ductility:

Copper:

In order to establish the simplest mode of tensile deformation behavior subsequent to tensile or torsional prestrain[7] without the complication of transition temperature, the initial tests were conducted with soft ductile copper (Fig. 4). The tensile ductility for zero prestrain at a test temperature of −321 F was approximately equal to that at a test temperature of +75 F. The linear type curves described earlier (Figs. 1(*a*) and 1(*c*)) for retained tensile ductility *versus* tensile prestrain are shown for copper in Fig. 4.

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[7] All of the tensile and torsional prestraining operations in this investigation were performed at room temperature (+75 F).

The curves for retained tensile ductility, ϵ_f,[8] *versus* torsional prestrain at test temperatures of +75 F and −321 F were quite similar to one another. As shown schematically in Fig. 1(*b*), with increasing torsional prestrain, the retained tensile ductility first decreased at a rather slow and essentially linear rate (the subcritical branch of the curve) until a critical prestrain was reached. Thereupon, with further increase in prestrain, there was a rapid decrease in the retained ductility (the supercritical branch of the curve). The introduction of the critical magnitude of torsional prestrain into the test specimen was accompanied by a change in the tensile fracture appearance from one which for less than critical magnitudes of torsional prestrain is of a "cup-cone" type to one of a helical type, Swift's (4) so-called "wolf-ear" fracture. Finally, with further prestrain the curve became approximately horizontal (the terminal branch of the curve) and remained so up to the point of fracture in torsional prestrain (exhaustion of the ductility of the metal in torsion). The helical type of fracture continued to persist in the tension test up to this point.

For the copper at each given test tem-

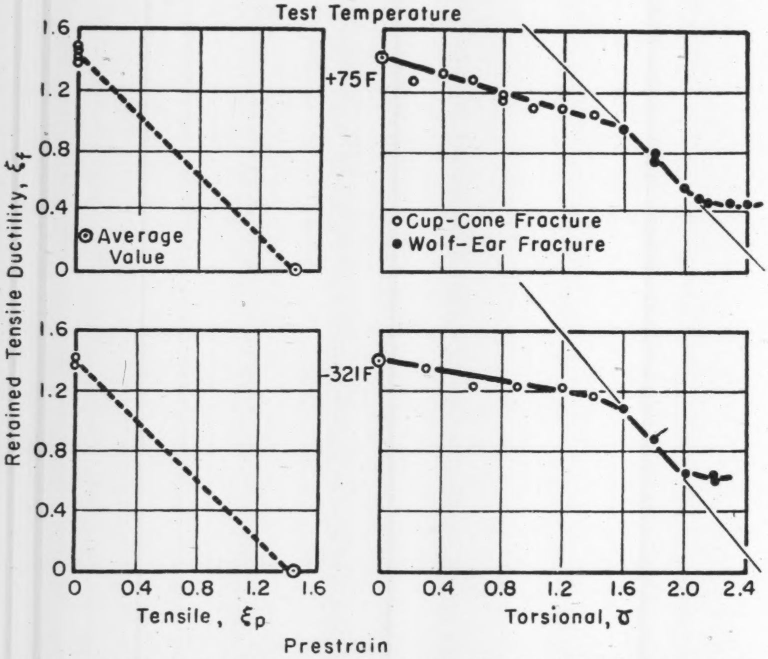

FIG. 4.—Effect of Test Temperature upon the Retained Tensile Ductility of Annealed Electrolytic Tough Pitch Copper Subsequent to Tensile or Torsional Prestrain (+75 F).

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[8] $\epsilon_f = \ln \frac{\text{Area After Prestrain}}{\text{Final Area}}$.

perature the critical shear strain required to initiate a helical "wolf-ear" tensile fracture was approximately $\gamma = 1.6$. The subcritical branch of the curve extended from $\gamma = 0.0$ to $\gamma = 1.6$; the supercritical branch of the curve extended from $\gamma = 1.6$ to approximately $\gamma = 2.0$; and the terminal branch extended from approximately $\gamma = 2.0$ to approximately $\gamma = 2.5$.

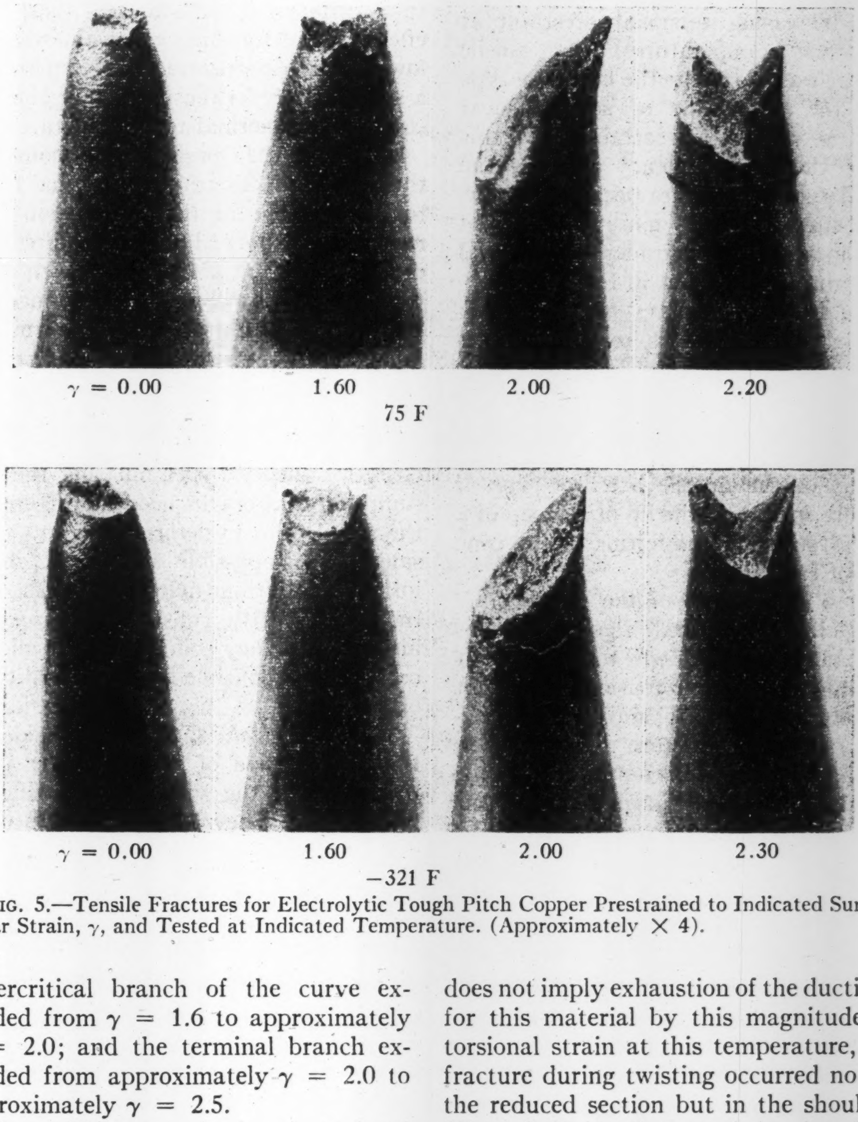

FIG. 5.—Tensile Fractures for Electrolytic Tough Pitch Copper Prestrained to Indicated Surface Shear Strain, γ, and Tested at Indicated Temperature. (Approximately × 4).

The critical magnitude of torsional prestrain necessary to cause the rapid decrease in retained tensile ductility and the helical type tensile fracture is independent of test temperature.

It should be noted that the maximum torsional prestrain obtainable ($\gamma = 2.5$) for copper at +75 F in this investigation does not imply exhaustion of the ductility for this material by this magnitude of torsional strain at this temperature, for fracture during twisting occurred not in the reduced section but in the shoulder fillet of the torsion specimen (see Fig. 3(*b*)) where the stress concentration factor was of the magnitude $K = 1.3$ (10).

Helical or "Wolf-Ear" Fracture:

Tensile fractures at test temperatures of +75 F and −321 F for specimens of the annealed copper torsionally prestrained to various magnitudes of surface shear strain, γ, are shown in Fig. 5. With increasing torsional prestrain at both test temperatures, the tensile fracture was of the ductile cup-cone type until the magnitude of surface shear strain $\gamma = 1.60$ was attained. At this value, the first tendency towards the helical type of fracture became evident. This effect became more pronounced with increased magnitudes of torsional prestrain, as is evident in Fig. 5.

The fracture surfaces of the copper tension test specimens torsionally prestrained to critical ($\gamma = 1.60$) or greater than critical ($\gamma > 1.60$) magnitudes of surface shear strain are essentially crystalline or brittle in nature, as contrasted to the ductile or silky type of fracture surface at the lip of the cup of a tension specimen exhibiting a cup-cone type of fracture.

Upon comparison of the tensile fractures, it is observed that specimens torsionally prestrained to equivalent amounts but tested in tension at different temperatures (+75 F and −321 F) exhibit similar fracture appearances, whether fractures of the cup-cone type or helical "wolf-ear" type. The tensile ductilities at zero prestrain for the face-centered-cubic copper were almost identical at test temperatures of +75 and −321 F.

Stimulated by Griffith's (11) microcrack theory in which he considered stress concentrations at the edges of submicroscopic cracks as the cause of the lowering of the cohesive strength in brittle materials from a high theoretical value, a number of possible explanations of the helical tensile fractures for normally ductile metals subsequent to pretorsion have arisen.

Swift (4) deduced that the helical "wolf-ear" tensile fracture was a manifestation of a shear type failure because of the delineation of the plane of maximum shear in tension (45 deg with respect to the tensile axis) by the fracture surface. He postulated that the effect of the torsional prestrain was to lower the shear stress required to produce a shear failure in tension rather than a conventional normal tensile fracture.

Gensamer (12) preferred to consider the "wolf-ear" fracture as a normal fracture by postulating that pre-torsion had reduced the normal stress required to cause fracture on a 45-deg plane so that the specimen exhibited a preference to fracture on a plane subjected to the maximum normal stress in torsion rather than on one subjected to the maximum normal stress in tension.

Zener and Hollomon (13) extended Griffith's theory by eliminating the assumption that cracks need be present in the metal prior to deformation and considered such possible sources of crack formation during deformation as the fracture of brittle constituents or weak interphases. They concluded that microcracks are responsible for the helical type of fracture.

Backofen *et al.* (5, 6) have considered the helical type of fracture after pretorsion from the viewpoint of aligned microcracks. They postulated that after the severe reduction of a metal by rolling, forging, etc., into a bar, all the cracks will be aligned parallel to the direction of working. During the process of twisting a specimen parallel to this direction, they considered that the cracks will be realigned at an angle ϕ with respect to the specimen axis, so that the torsional shear strain at the surface, γ, will be

$$\gamma = \tan \phi = \frac{R\theta}{L}$$

where R is the distance of the crack from the specimen axis and $\frac{\theta}{L}$ is the angle of twist in radians per unit length.

Effective Strain:

In Fig. 4, for both test temperatures (+75 and −321 F) the supercritical branch of each torsional prestrain curve extrapolated to the abscissa axis to a value of torsional shear strain of approximately $\gamma = 2.48$. On the other hand, each of the retained tensile ductility curves (+75 F and −321 F) for tensile prestrain possessed a terminal point on the abscissa axis equal to $\epsilon_p = 1.43$ (the tensile ductility of the copper at zero prestrain at +75 F). It seemed reasonable, then, that the first step in the attempt to correlate the effects of tensile and torsional prestrain upon the retained tensile ductility involved the further consideration of these values ($\epsilon_p = 1.43$ and $\gamma = 2.48$).

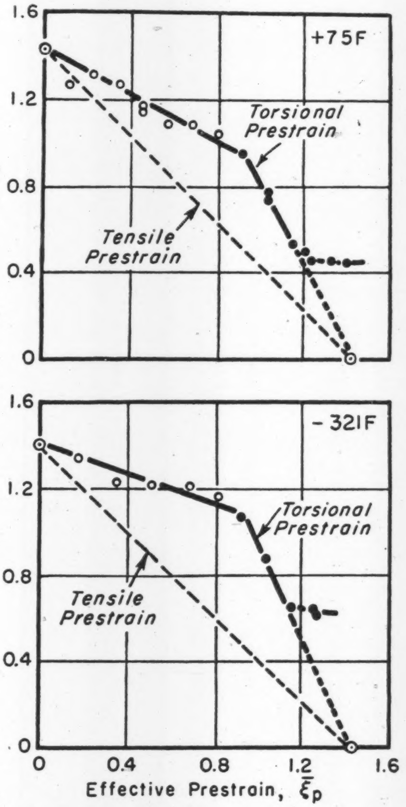

FIG. 6. Effect of Test Temperature Upon the Retained Tensile Ductility of Annealed Electrolytic Tough Pitch Copper Subsequent to Effective Prestrain (+75 F).

In order to effect the above correlation, consideration was given to the generalized or effective stress-strain relations (14):

$$\bar{\sigma} = \sqrt{\tfrac{1}{2}[(\sigma_1 - \sigma_2)^2 + (\sigma_2 - \sigma_3)^2 + (\sigma_3 - \sigma_1{}^2]}$$

$$\bar{\epsilon} = \sqrt{\tfrac{2}{3}(\epsilon_1{}^2 + \epsilon_2{}^2 + \epsilon_3{}^2)}$$

where σ_1, σ_2, and σ_3 are the true principal stresses; ϵ_1, ϵ_2, and ϵ_3 are the true principal strains; $\bar{\sigma}$ is the effective stress and $\bar{\epsilon}$ is the effective strain.

From the above equation for strain:

$\bar{\epsilon}_p = \epsilon_p$ for tensile prestrain,

$\bar{\epsilon}_p = \gamma/\sqrt{3}$ for torsional prestrain,

$\bar{\epsilon}_f = \epsilon_f$ for retained tensile ductility.

Justification for employing this form of effective strain is based on the results of Jackson (15) and Lankford (16) who showed that if prestrain under a stress state different from tension was plotted in terms of effective strain, the composite stress-strain curve coincided with one obtained for simple tension alone.

Figure 6 shows such a plot in terms of effective prestrain for copper for test temperatures of +75 F and −321 F. At each temperature the supercritical branch (neglecting the terminal branch) of the curve for torsional prestrain extrapolates to the same value of effective prestrain as the terminal point on the abscissa axis for the tensile prestrain curve.

Several curves showing the effect of torsional prestrain at room temperature upon the retained tensile ductility at room temperature (in terms of reduction in area) have been taken from the literature (4, 5, 17) and replotted in terms of effective prestrain and effective retained tensile ductility in Fig. 7.

Although for both the electrolytic tough pitch copper (5) and the annealed commercial SAE 4340 steel (17) the supercritical branch of the curve extrapolates to the same value of effective prestrain as the tensile ductility at zero prestrain of the respective metal, this is not the case for the annealed mild steel (4). No explanation can be made at this time for the non-conformity of this latter steel.

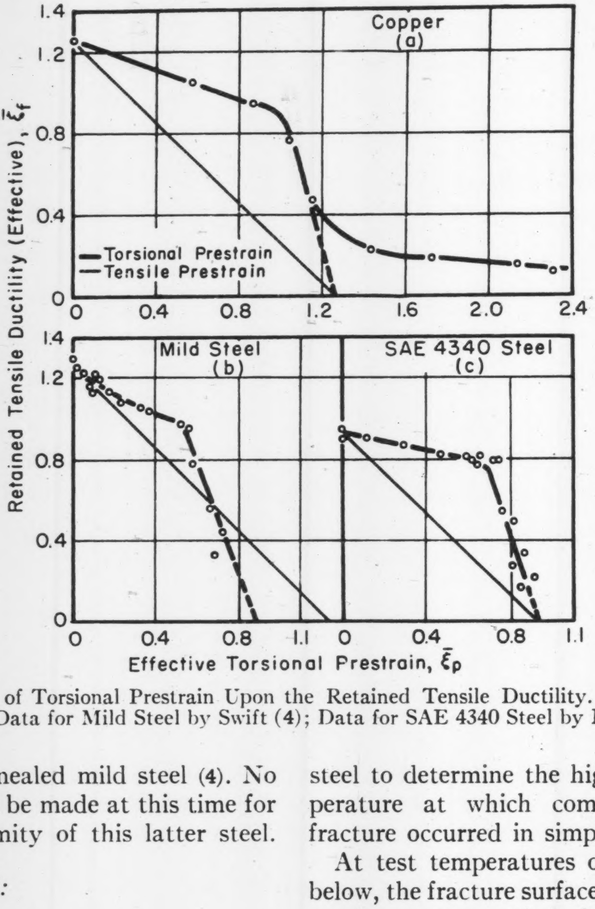

FIG. 7.—Effect of Torsional Prestrain Upon the Retained Tensile Ductility. Data for Copper by Backofen (5); Data for Mild Steel by Swift (4); Data for SAE 4340 Steel by Fields (17).

SAE 1020 Steel:

Transition Temperature:

Tension test specimens of spheroidized SAE 1020 steel were tested in tension at various temperatures in the range +75 to −321 F. The experimental curves for retained tensile ductility, ϵ_f, *versus* test temperature and for tensile fracture stress s_f,[9] *versus* test temperature are shown in Fig. 8.

Unfortunately, there is no narrow temperature range on the retained ductility curve which defines the transition temperature. However, by examination of the fractured specimens, it was possible by observations similar to those made by Eldin and Collins (18) on a mild steel to determine the highest test temperature at which completely brittle fracture occurred in simple tension.

At test temperatures of −260 F and below, the fracture surfaces of the tension specimens were entirely brittle and crystalline in appearance. However, at a test temperature of −240 F the fracture surface contained at its center a dark, noncrystalline appearing, fibrous circle ap-

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[9] $s_f = \dfrac{\text{Load at Fracture}}{\text{Fracture Area}}$.

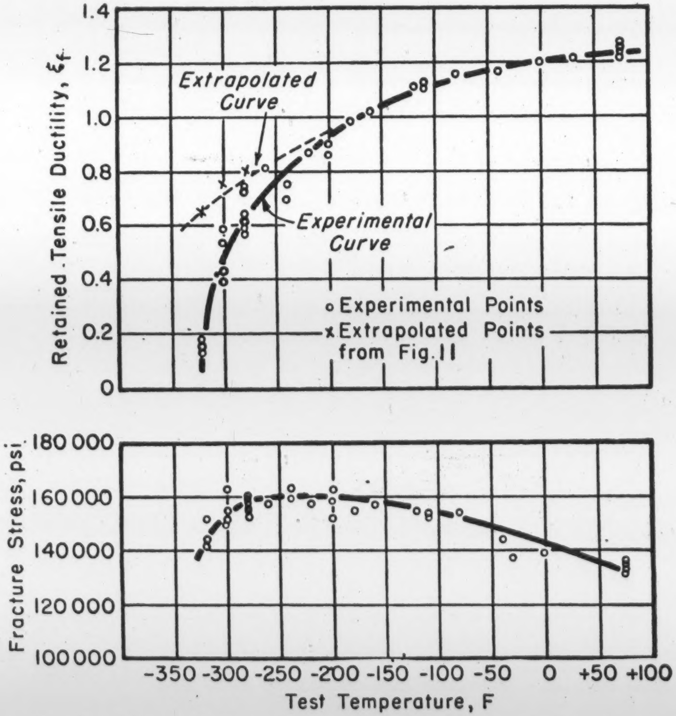

FIG. 8.—Effect of Test Temperature upon the Tensile Fracture Properties of Spheroidized SAE 1020 Steel.

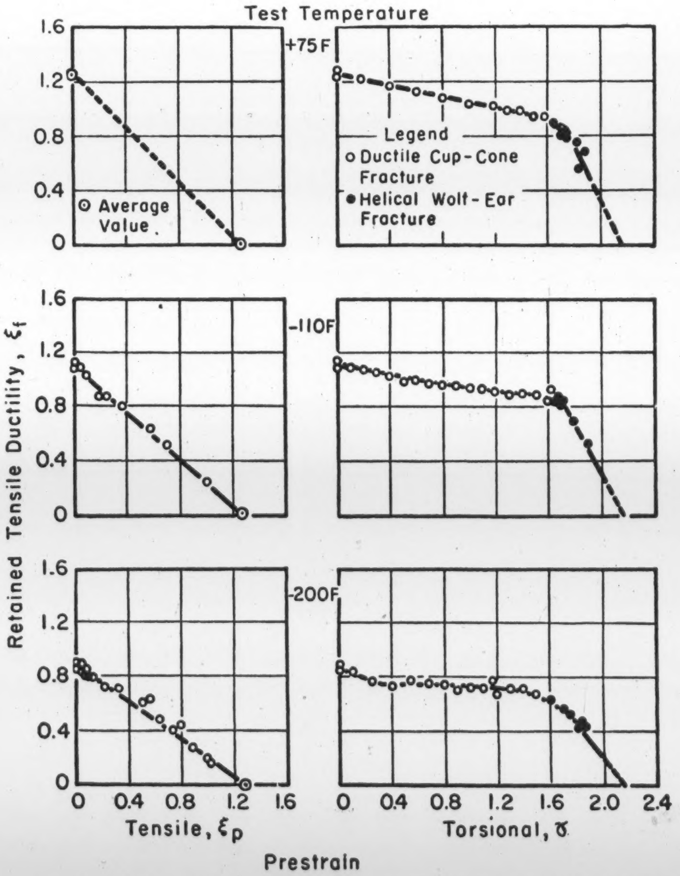

FIG. 9.—Effect of Test Temperature upon the Retained Tensile Ductility of Spheroidized SAE 1020 Steel Subsequent to Tensile or Torsional Prestrain (+75 F).

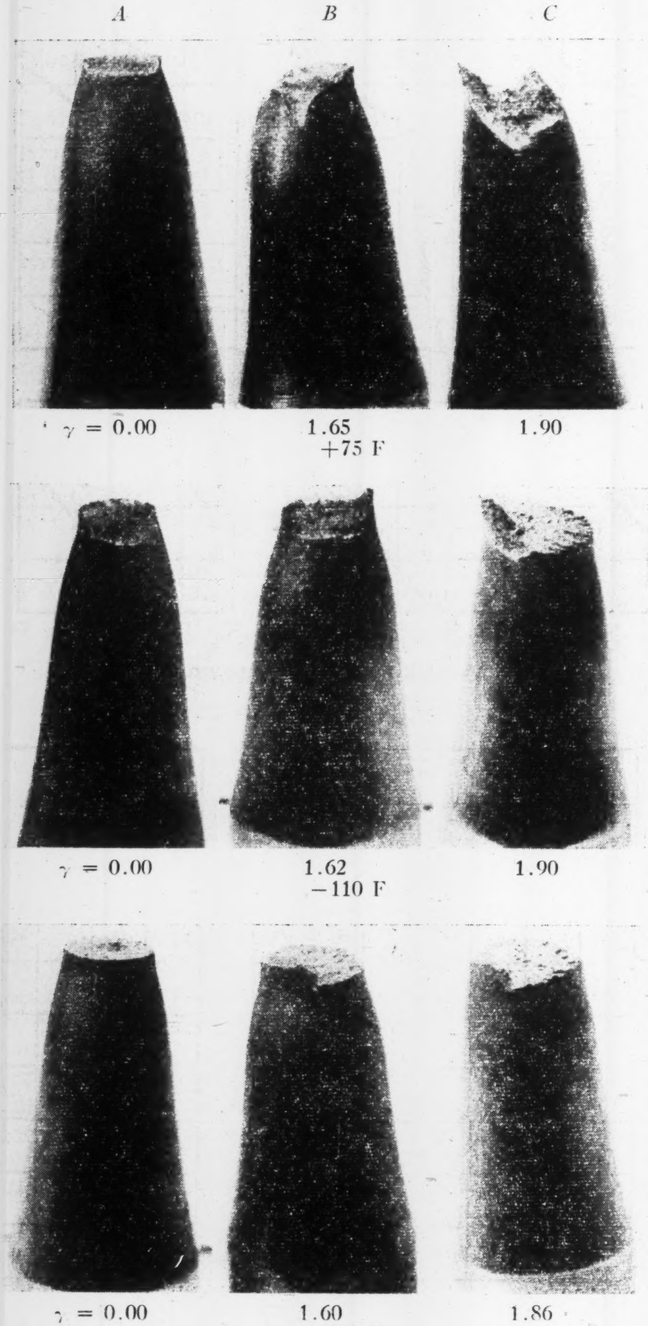
A
B
C
γ = 0.00
1.65
+75 F
1.90
γ = 0.00
1.62
−110 F
1.90
γ = 0.00
1.60
−200 F
1.86

FIG. 10.—Tensile Fractures for Spheroidized SAE 1020 Steel Prestrained to Indicated Surface Shear Strain, γ, and Tested at Indicated Temperature. (Approximately × 4).

proximately 2 to 3 mm in diameter, the remainder of the fracture surface retaining the crystalline appearance characteristic of lower test temperature fractures. With increasing test temperature, the size of this dark dot increased at the expense of the brittle portion of the fracture surface until at a test temperature of −120 F, the black circle covered the whole fracture surface, that is, the fracture was now entirely of the ductile cup-cone type. It was concluded that any test temperature at which there was visual evidence of a fibrous portion in the fracture surface was above the transition temperature of the steel. Hence, the transition temperature for the SAE 1020 steel was estimated to be −250 F.

It is also interesting to note that the tensile fracture stress attains its maximum value at this test temperature.

Effect of Tensile or Torsional Prestrain at Higher Than Transition Test Temperatures:

After tensile or torsional prestrain of the SAE 1020 steel at +75 F, subsequent tension tests were performed at three different temperatures above the transition temperature (75, −110, and −200 F).

The curves for retained tensile ductility, ϵ_f, *versus* prestrain in tension, ϵ_p, or torsion, γ, at the three test temperatures are shown in Fig. 9. As expected, the retained ductility *versus* tensile prestrain curve for each of these test temperatures is a straight line. The terminal point on the abscissa axis is equal to the tensile ductility ($\epsilon_p = 1.26$) at the prestrain temperature; the terminal point on the ordinate axis is equal to the tensile ductility at the test temperature, that is, $\epsilon_f = 1.26 + 75$ F; $\epsilon_f = 1.12$ at −110 F; and $\epsilon_f = 0.92$ at −200 F.

At the same test temperatures, the curves for retained tensile ductility *versus* torsional prestrain, Fig. 9, are similar to those obtained for the copper (Fig. 4) used in this investigation. However, for the SAE 1020 steel, even at a test temperature of +75 F, there is no terminal branch to the curve as for copper. This factor may be due to a lack of sufficient ductility in torsion, that is, the maximum surface shear strain obtained experimentally by torsional prestrain for the steel was approximately $\gamma = 1.9$. The nearly linear subcritical branch of each curve extends to an approximate value of $\gamma = 1.6$ to 1.7. The supercritical branch of the torsional prestrain curve for each of the given different test temperatures extrapolates to the same value $\gamma = 2.17$ on the abscissa (torsional prestrain) axis.

Helical "Wolf-Ear" Fracture:

Figure 10 shows fractures of specimens of SAE 1020 steel torsionally prestrained at +75 F to various magnitudes of surface shear strain and subsequently tested in tension at the temperatures of +75, −110, and −200 F.

The photographs in column *A* of Fig. 10 show the change in fracture appearance with test temperature at zero prestrain ($\gamma = 0.00$). Although the fractures for test temperatures of +75 and −110 F are of the ductile cup-cone type, the fracture at −200 F tends to be more brittle in nature and exhibits the small dark fibrous circle at its center as previously described. These photographs are the prototypes of fractures for tension test specimens torsionally prestrained to less than critical magnitudes of surface shear strain.

The photographs in Column *C* of Fig. 10 show fractures obtained for tension specimens torsionally prestrained to greater than critical magnitudes of surface shear strain. It is noted that with decreasing test temperature, the appearance of the helical "wolf-ear" fracture tends to become less pronounced. This change with decreasing test temperature is detectable with somewhat less ease for the fractured tension test specimens torsionally prestrained to critical magnitudes ($\gamma = 1.6$ to $\gamma = 1.7$), as shown in column *B* of Fig. 11.

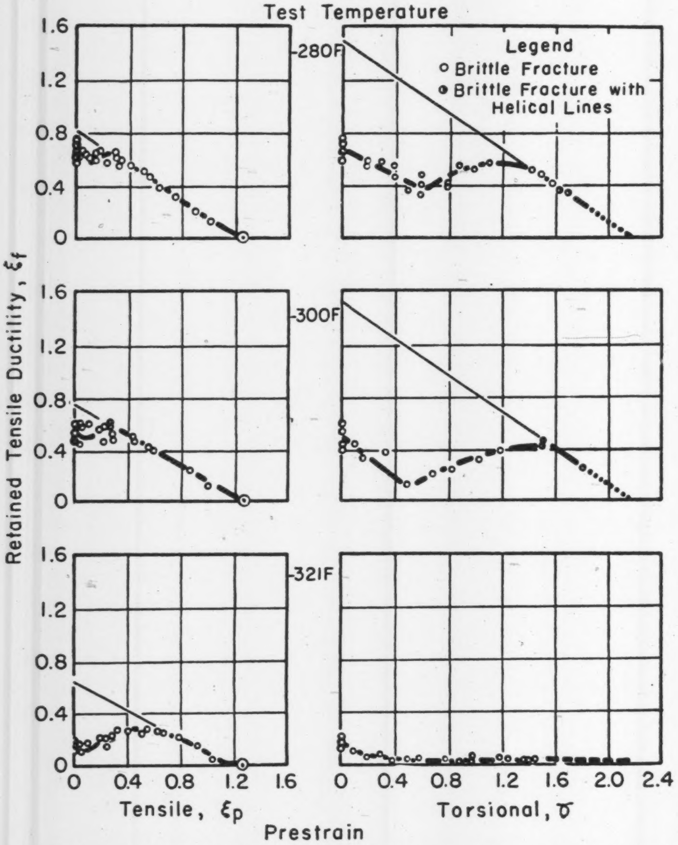

FIG. 11.—Effect of Test Temperature on the Retained Tensile Ductility of Spheroidized SAE 1020 Steel Subsequent to Tensile or Torsional Prestrain (+75 F).

The "wolf-ear" or helical type tensile fractures obtained at different test temperatures subsequent to equal magnitudes of greater than critical torsional prestrain are quite similar when the tensile ductilities at zero prestrain are approximately equal (see Fig. 5 for copper). But they are quite different when the tensile ductilities at zero prestrain vary (see Fig. 10 for SAE 1020 steel).

Effect of Tensile and Torsional Prestrain at Lower Than Transition Test Temperatures:

The curves for SAE 1020 steel for retained tensile ductility, ϵ_f, *versus* prestrain in tension, ϵ_p, or torsion, γ, at the three lower than transition test temperatures (−280, −300, and −321 F) are shown in Fig. 11.

For tensile prestrain, the curves are of the type obtained by Ripling and Baldwin (3) for an annealed steel. The basic three branches—the metastable, the transition and the stable branches—were obtained for each curve at each of the three test temperatures, shown schematically in Fig. 1(*d*). With decreasing test temperature, the amount of tensile prestrain necessary to attain a minimum in the curve decreases and the prestrain necessary to attain a maximum in the curve increases.

The stable branch of the tensile prestrain curve at each lower than transition test temperature extrapolates to the ordinate axis to a value of retained ductility which falls on the curve obtained by extrapolating the supertransition part of the retained tensile ductility *versus* test temperature curve in Fig. 8. For each of these test temperatures, the difference in retained ductility between the extrapolated curve and the experimental curve denotes that part of the low-temperature ductility deficiency which is rheotropic in nature.

The curves for retained tensile ductility *versus* torsional prestrain at test temperatures of −280 and −300 F resemble those for tensile prestrain in that each is composed of the three usual parts, that is, the metastable, transition, and stable branches. Again, with decreasing test temperature, the amount of torsional prestrain necessary to attain a minimum in the curve decreases (including the −321 F test temperature), and the torsional prestrain necessary to attain a maximum increases.

It was not possible to obtain data for specimens prestrained in torsion to surface shear strains greater than 1.45 and tested in tension at −321 F because all such specimens fractured during the tension test at points along the axis of the tension specimen which were outside the minimum section.

Fracture Appearance at Lower Than Transition Test Temperatures:

All of the tensile fractures obtained after torsional prestrain at test temperatures of −280, −300, and −321 F were of the brittle crystalline type (no fibrous dark dot at the center of the fracture surface).

For a test temperature of −280 F, helical markings were present on the external surface of fractured tension test specimens subsequent to a torsional surface shear strain greater than or equal to 1.65, but no "wolf-ear" fractures, *per se*, were observed. For a test temperature of −300 F, these helical markings were obtained on fractured tension test specimens torsionally prestrained to a surface shear strain greater than or equal to 1.55.

But for a test temperature of −321 F, not only no helical "wolf-ear" fractures but also no helical surface markings were observed for any of the fractured tension test specimens previously prestrained in torsion up to the experimental limit $\gamma = 1.45$. Upon consideration of the surface shear strain gradient along the axis of the tension test specimen (see Fig. 3) after torsional prestrain, the reduction in section size at the center (of length) of the tension test specimen may have been of insufficient magnitude to prevent the specimen from fracturing at another point along the specimen axis at extremely low test temperatures.

Retained Tensile Ductility Versus Effective Prestrain:

Figure 12 shows curves for SAE 1020 steel of retained tensile ductility (effective) *versus* effective prestrain ($\bar{\epsilon}_p = \epsilon_p$ for tensile prestrain and $\bar{\epsilon}_p = \gamma/\sqrt{3}$ for torsional prestrain) at the three lower than transition and three higher than transition test temperatures used in this investigation. Since $\bar{\epsilon}_p = \epsilon_p$, the curves for tensile prestrain are the same as those shown in Figs. 9 and 11.

The torsional prestrain curves in Fig. 12 are similar to those shown in Figs. 9 and 11 but are essentially compressed along the abscissa axis for $\bar{\epsilon}_p = \gamma/\sqrt{3}$. On the basis of effective prestrain, the terminal point on the abscissa axis for the stable branch of the tensile prestrain curve is identical to the terminal point on the abscissa axis for the extrapolated stable branch of the torsional prestrain curve at each of the test temperatures. However, although the curves for tensile and torsional prestrain at the lower than transition temperatures are somewhat similar in shape (except for −321 F), the maxima and minima in the curves obtained for each mode of prestrain do not correspond in magnitude of effective prestrain, nor do the retained tensile ductilities obtained under these conditions correspond in magnitude.

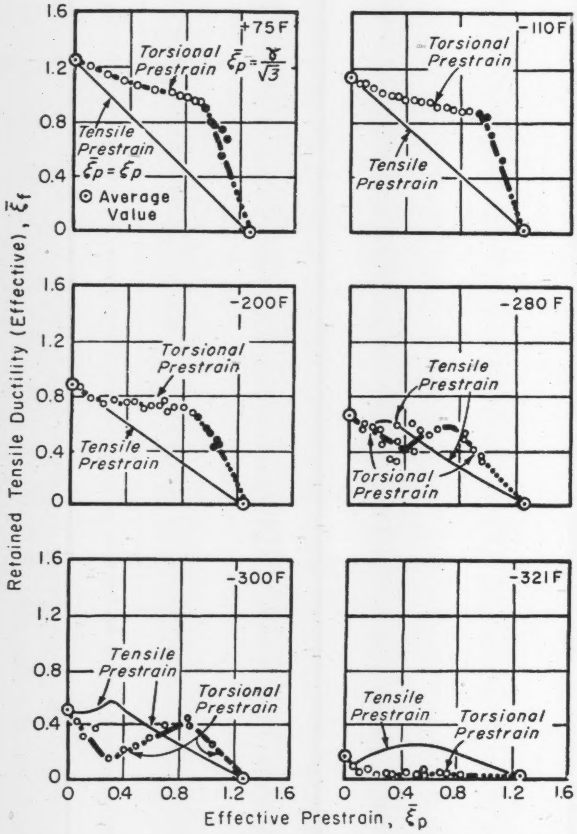

FIG. 12.—Effect of Test Temperature on Retained Tensile Ductility of Spheroidized SAE 1020 Steel Subsequent to Tensile or Torsional Prestrain (+75 F).

Rheotropic Embrittlement:

For test temperatures of −280 and −300 F (Fig. 11) the stable branches of the retained tensile ductility *versus* torsional prestrain curves both extrapolate to values of approximately $\epsilon_f = 1.5$ on the ordinate (retained tensile ductility) axis. This extrapolated value is of greater magnitude than the tensile ductility of the SAE 1020 steel at +75 F. It is possible that the slope of the stable branch of the ductility *versus* torsional prestrain curve at test temperatures of −280 and −300 F may have been influenced by the steep slope of the supercritical branch of the ductility (+75 F) *versus* torsional prestrain curve (Fig. 11). This influence could explain the difference between the extrapolated ductility values for the stable branches of the ductility *versus* tensile prestrain and ductility *versus* torsional prestrain curves for each of the above test temperatures.

The stable branch of each curve (Fig. 11) at the given test temperatures extrapolates to $\gamma = 2.17$ on the abscissa (torsional prestrain) axis. This value is identical with the extrapolated value of the supercritical branch of the retained tensile ductility *versus* torsional prestrain curves for the spheroidized SAE 1020 steel at the test temperatures, +75, −110, and −200 F, in Fig. 9.

For lower than transition test temperatures, matching up the terminal points by means of effective prestrain (Fig. 12) for each test temperature does not imply point for point coincidence of the curves for different modes of prestrain. It appears that the positions and magnitudes of the maxima and minima are at least in part a function of the prestrain stress state.

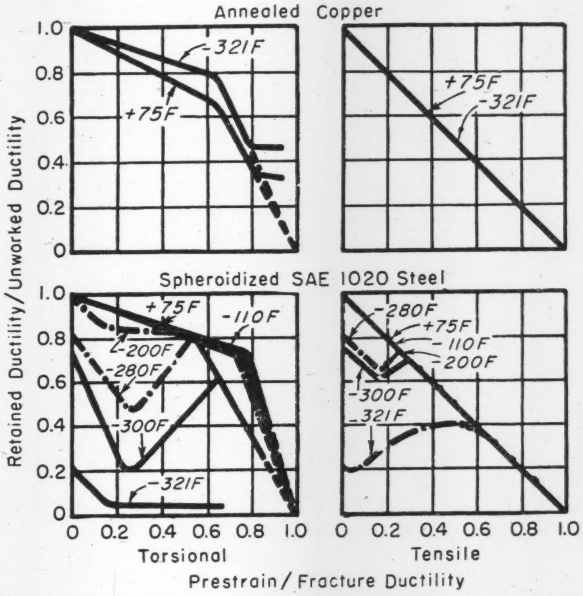

FIG. 13.—Effect of Test Temperature on Tensile Ductility Subsequent to Torsional or Tensile Prestrain.

A reduced type of plot is shown in Fig. 13 for copper (face centered cubic) and for SAE 1020 steel (body centered cubic) for all test temperatures used in this investigation. Here, for each material at each temperature, the ratio of the retained ductility to the ductility at zero prestrain is plotted *versus* the ratio of the effective (torsional or tensile) prestrain to the fracture ductility.

Similarities between curves here are evident. The supercritical branch of the torsional prestrain curve for each of the above metals extrapolates to the abscissa axis to the same value (1.0 on the reduced plot) as the terminal point on the abscissa axis for the tensile prestrain curve. The torsional prestrain curves for the copper and for the SAE 1020 steel at higher than transition test temperatures are relatively alike in shape.

For the steel at the lower than transition test temperatures of −280 and −300 F, each curve for tensile or torsional prestrain consists of the requisite three branches: metastable, transition, and stable.

For both tensile and torsional prestrain at lower than transition test temperatures, the magnitude of reduced prestrain necessary to attain a minimum in the curve decreases with decreasing test temperature, whereas the magnitude of reduced prestrain necessary to attain a maximum in the curve increases with decreasing test temperature.

The slope of the stable branch of each curve (−280 and −300 F) for torsional prestrain is intermediate between the slope (45 deg) of the stable branch of each curve (−280 and −300 F) for tensile prestrain and the very steep slope of the supercritical branch of the curve obtained for a higher than transition test temperature (+75, −110, or −200 F).

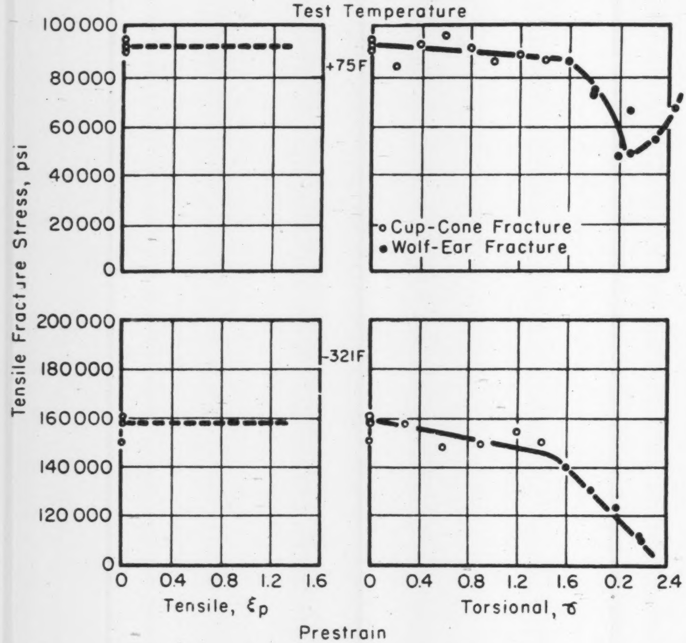

FIG. 14.—Effect of Test Temperature on Tensile Fracture Stress of Annealed Electrolytic Tough Pitch Copper Subsequent to Tensile or Torsional Prestrain (+75 F).

As previously stated, there is an elimination of the "wolf-ear" tensile fracture for torsional prestrain at lower than transition test temperatures. It appears that the type of tensile fracture obtained at any test temperature subsequent to critical or greater than critical magnitudes of surface shear strain is a result of the competitive effort between (*a*) the tendency towards a "wolf-ear" helical type fracture and (*b*) the tensile fracture normally obtained for the non-prestrained metal at the given test temperature.

Thus at higher than transition test temperatures, the tensile ductility at zero prestrain is high and the "wolf-ear" fracture manifests itself in its complete form. At lower than transition test temperatures, the tensile ductility at zero prestrain is low and hence the low-temperature embrittling effect overshadows the tendency towards a "wolf-ear" fracture—the only manifestation of "wolf-ear" fracture tendency consists of helical surface markings on the fractured tension specimen. At any intermediate test temperature, the final fracture appearance would be a compromise between these extremes.

Tensile Fracture Stress:

Although the majority of emphasis in this investigation was placed upon a correlation of the effects of tensile and torsional prestrain upon the retained tensile ductility of copper and SAE 1020 steel, some consideration was given to a comparison of the effects of tensile and torsional prestrain upon the other tensile fracture property, the tensile fracture stress.

Copper:

Figure 14 shows the effects of tensile and torsional prestrain upon the tensile fracture stress for annealed copper at test temperatures of +75 and −321 F.

For a test temperature of +75 F, the fracture stress remains constant with increasing tensile prestrain. When the prestrain stress state is changed to torsion, the fracture stress curve becomes more complex. With increasing torsional prestrain, the fracture stress decreases very slightly for less than critical magnitudes of torsional prestrain. At greater than critical magnitudes of torsional shear strain, the presence of the helical "wolf-ear" fracture is accompanied by a rapid decrease in fracture stress (4) until a torsional prestrain equal in magnitude to the beginning of the terminal branch of the retained ductility *versus* torsional prestrain curve is reached (Fig. 4). Thereupon the fracture stress continues to increase with increasing torsional prestrain.

At a test temperature of −321 F, the fracture stress curves for tensile and torsional prestrain are quite similar to those described above for a test temperature of +75 F, except that there is no minimum exhibited in the fracture stress *versus* torsional prestrain curve.

It is evident that not only is the attainment of the helical "wolf-ear" tensile fracture accompanied by a decrease in retained tensile ductility but also by a rapid decrease in tensile fracture stress.

SAE 1020 Steel:

Figures 15 and 16 show the effects of tensile and torsional prestrain upon the tensile fracture stress for SAE 1020 steel at the three higher than transition test temperatures and the three lower than transition test temperatures, respectively.

At +75 and −110 F, the tensile fracture stress remains constant with increasing tensile prestrain. With increasing torsional prestrain at both of these test temperatures, the fracture stress increases slightly until at approximately the critical magnitude of torsional prestrain the fracture stress decreases rapidly with increasing prestrain.

At a test temperature of −200 F, increasing tensile prestrain causes a slight linear increase in the tensile fracture stress. However, for increasing torsional

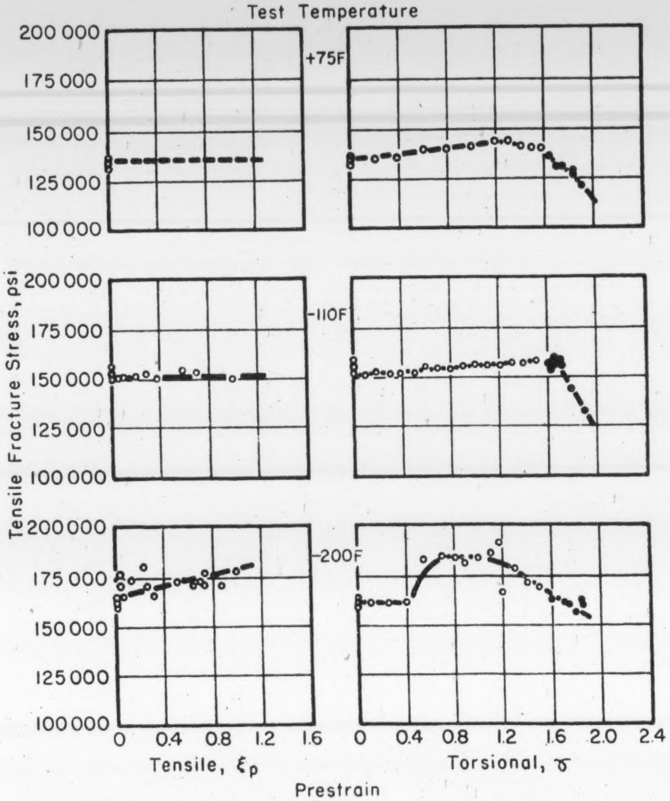

FIG. 15.—Effect of Test Temperature on the Tensile Fracture Stress of Spheroidized SAE 1020 Steel Subsequent to Tensile or Torsional Prestrain.

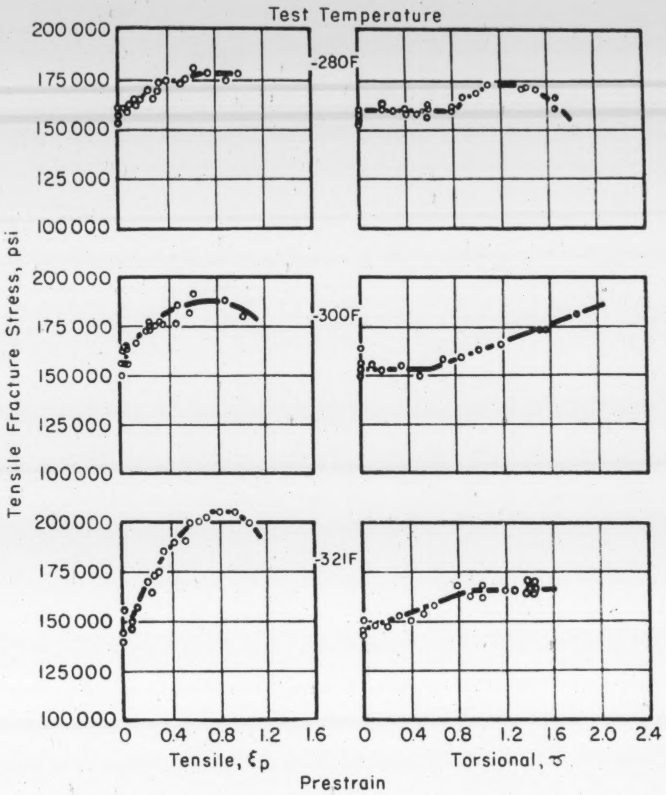

FIG. 16.—Effect of Test Temperature on the Tensile Fracture Stress of Spheroidized SAE 1020 Steel Subsequent to Tensile or Torsional Prestrain.

prestrain the fracture stress at −200 F remains constant for an interval of prestrain, then attains a maximum value and finally continues to decrease with increasing torsional prestrain. The appearance of the helical "wolf-ear" fracture at this test temperature does not cause any change in shape of the fracture stress *versus* torsional prestrain curve.

The tensile fracture stress *versus* tensile prestrain curves at the test temperatures of −280, −300, and −321 F are all quite similar (Fig. 16). With increasing tensile prestrain, the fracture stress increases rather rapidly, reaches a maximum value, and then starts to descend.

At a test temperature of −300 F, the fracture stress remains constant for an interval of torsional prestrain and then continually increases in a linear manner with increasing torsional prestrain. But at a test temperature of −321 F, the fracture stress increases immediately with increasing torsional prestrain, and then with further increased prestrain the curve eventually flattens out.

It should be noted from Figs. 15 and 16 that the appearance of the helical "wolf-ear" fracture assumes continually less importance with decreasing test temperature in influencing the shape of the tensile fracture stress *versus* torsional prestrain curve at a given test temperature.

CONCLUSIONS

A correlation of the effects of tensile and torsional prestrain upon the tensile fracture properties for several metals at various test temperatures has indicated that for:

A. *Copper:*

1. The curves for retained tensile ductility (+75 and −321 F) *versus* prestrain were quite dissimilar in shape when the prestrain stress state was changed from torsion to tension.

2. The terminal points of the curves on the prestrain and retained ductility axes may be matched (after extrapolation of the supercritical branch of the torsional curve to the prestrain axis) by considering the torsional and tensile prestrain in terms of effective or generalized prestrain.

3. Tension test specimens torsionally prestrained equivalent amounts exhibited similar fracture appearances at different test temperatures, whether the fracture was of the cup-cone or helical "wolf-ear" type. The "wolf-ear" tensile fracture was obtained after a critical magnitude of torsional prestrain which was the same for both test temperatures.

4. The attainment of the helical tensile fracture subsequent to critical or greater than critical magnitudes of torsional prestrain was, in general, accompanied by a severe decrease in retained tensile ductility and fracture stress.

B. *SAE 1020 Steel:*

1. For those test temperatures above the transition temperature, the helical "wolf-ear" tensile fracture is obtained subsequent to critical or greater than critical magnitudes of torsional prestrain. With decreasing test temperature the "wolf-ear" fracture characteristics became less pronounced.

2. At test temperatures below the transition temperature for the steel the "wolf-ear" type of tensile fracture essentially disappeared, being manifested only in helical surface markings on the broken tension test specimen.

3. The degree of manifestation and the appearance of the helical "wolf-ear" fracture itself were dependent upon the tensile ductility for zero prestrain at the given test temperature.

4. The type of tensile fracture appearance obtained at any test temperature subsequent to a critical or greater than

critical magnitude of torsional prestrain was an effective compromise between two factors: (*a*) the tendency towards the helical "wolf-ear" type of fracture and (*b*) the tensile fracture normally obtained for the non-prestrained metal at the given test temperature.

5. The critical magnitude of torsional prestrain necessary to attain the helical type of tensile fracture was relatively independent of test temperature.

6. Although there was rheotropic behavior in tension evidenced for both tensile and torsional prestrain, the stable branches of the retained tensile ductility curves for each type of prestrain did not extrapolate to the same value of retained tensile ductility.

7. When effective prestrain was considered, the retained tensile ductility curves for the two types of prestrain matched only at the terminal points for a given test temperature.

8. Variation in prestrain stress state generally caused considerable variation in the shape of the curves for tensile fracture stress *versus* prestrain.

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APPENDIX

DETAILS OF MATERIAL AND PROCEDURE

Material:

All material used in this investigation was received in the form of $\frac{3}{4}$-in. diameter hot rolled rods. The chemical analyses for the copper and steel used are shown in Table I.

All of the rods of SAE 1020 steel used in this investigation were rolled from a single billet of aluminum-killed steel.

Heat Treatment

Copper:

The copper rods were annealed at 900 F for 1 hr and then air cooled.

SAE 1020 Steel:

Rods of this material were annealed at 1700 F for $1\frac{3}{4}$ hr, furnace cooled to room temperature, spheroidized at 1300 F for 120 hr and finally furnace cooled.

SPECIMENS

The torsional prestrain specimens for all the materials used in this investigation were of the type shown in Fig. 3(*b*). The reduced section only of the specimen was actually twisted, the ends of the specimen being restrained in the jaws of the torsion machine.

Tension test specimens of the type shown in Fig. 3(*a*) were machined from the center of the reduced section of the prestrained torsion specimen.

All of the torsionally prestrained copper specimens were finished to a tension test specimen of a diameter of 0.212 in. at the minimum section, as were all tension test specimens of the spheroidized SAE 1020 steel tested at temperatures above −280 F.

At test temperatures below −280 F, tension test specimens of the spheroidized SAE 1020 steel had to be finish-machined to a diameter of 0.180 in. at the minimum section in order to avoid specimen shoulder fractures and to prevent damage to the tension testing equipment.

TABLE I. CHEMICAL ANALYSES, PER CENT.

Material	Carbon	Silicon	Manganese	Phosphorus	Sulfur	Copper	Iron
Copper	...	...	...	...	0.003	99.95	0.02
SAE 1020 steel	0.199	0.10	0.73	0.009	0.044	...	remainder

EQUIPMENT AND TEST PROCEDURE

A commercial type torsion machine with a 10,000 lb-in. maximum torque capacity was used to effect torsional prestrain. All torsional prestrains were performed at a strain rate of 120 deg per min.

A 60,000-lb capacity commercial type hydraulic tension testing machine was used for both tensile prestrain and for the subsequent tension test after either torsional or tensile prestrain. A specially constructed loading fixture (**19**) yielding an eccentricity of less than 0.001 in. was used for both the tensile prestraining and tension testing.

The time interval between the tensile prestrain and the tension test was about 4 hr; but when the prestrain was torsional in nature, the interval between prestrain and tension test was longer because of the time required to machine a tension test specimen from a specimen prestrained in torsion.

The magnitudes of tensile prestrain and of retained tensile ductility were obtained by means of diameter measurements made with a microcomparator prior to and subsequent to each particular strain.

The coolants used for the low-temperature tests were:

−40 F to −110 F	Isopentane and dry ice
−110 F to −240 F	Isopentane and liquid nitrogen
−260 F to −300 F	Nitrogen vapor from boiling liquid nitrogen
−321 F	Liquid nitrogen at boiling point

EFFECT OF BRITTLE SKINS ON THE DUCTILITY OF METALS*†

By G. W. Form[1] and W. M. Baldwin, Jr.[2]

Synopsis

Brittle skins (for example, carburized cases) embrittle an otherwise ductile metal to a greater degree than would be anticipated by the percentage of bulk that the skin occupies. Evidence is adduced to show that in tension tests on metals with brittle skins, the skin cracks at a negligible strain, and that from this point on the test is merely a notch tension test of the core. This points out, among other things, that notch-sensitive steels are ill-suited for carburizing purposes.

Many metals acquire brittle skins during processing. Carburized steels are one example. The literature contains many references to this problem, all of which suggest that a brittle skin is capable of embrittling a metal out of all proportion to the percentage of bulk that the skin itself occupies.

In 1921 Brearley (**1**)[3] reported that the impact toughness of a low-carbon steel is reduced to less than one-fifth of its original value when the steel is carburized. Zapffe and Haslem (**2**) observed a sharp decrease in bending angle of chromium-plated stainless steel wire in comparison with the bare wire. Wyatt and Grant (**3**) concluded that regardless of temperature and time of nitriding, nitrided titanium specimens were always brittle. Spencer (**4**) showed that nickel steels suffer a considerable loss in ductility after nitriding. Floe (**5**) reported a drop of reduction of area from 59.4 to 43.4 per cent when nitralloy was nitrided.

Moreover, some apparently brittle metals can be rendered ductile by pickling or grinding off relatively small thicknesses of the surface, as for example, tungsten (**6**), chromium (**6,7**), molybdenum (**6**), titanium (**8**), and hydrogen-embrittled steel (**9**). While Kroll (**6**) maintains that no conclusion can yet be reached as to the factors responsible for eliminating brittleness in the metals by pickling, other investigators (**7,8**) definitely associate the improvement in ductility with the removal of impurities in the surface. This implies, of course, that a contaminated metal surface exerts an embrittling effect.

Mention should be made here of the

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

† This paper is based on a portion of a research program ("The Effects of Stress Concentration and Triaxiality on the Plastic Flow of Metals") conducted in the Department of Metallurgical Engineering, Case Institute of Technology, in cooperation with the Office of Naval Research.

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[2] Research Professor, Case Institute of Technology, Cleveland, Ohio.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 658.

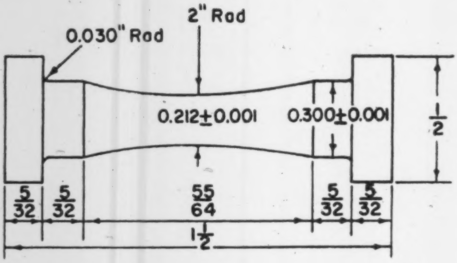

FIG. 1.—Standard Size Tensile Specimen Used in this Investigation.

thermal hardening (10–12) and the Rehbinder effect (13–15), since both phenomena originate from a contaminated surface. These phenomena are observed in single crystals only, however, and are disregarded in the following discussion where only polycrystalline aggregates are dealt with.

RESULTS AND DISCUSSION

Experimental Series A:

A general survey of the effects of brittle skins on otherwise ductile metals was made for (*a*) oxidized high-purity iodide titanium, (*b*) nitrided AISI 303 stainless steel, (*c*) hydrogen-embrittled tough-pitch copper, (*d*) chromium-plated copper, (*e*) carburized SAE 1020 steel, and (*f*) induction-hardened SAE 4340 steel.

TABLE 1.—SURFACE EMBRITTLING TREATMENTS.

Embrittling Process	Base Metal	Embrittlement Data		Maximum Case Depth, in.	Remarks
		Temperature, deg Fahr	Time of Exposure, hr		
Oxidation	High-purity-iodide titanium	1650	8	0.0045	Electrically heated furnace with an air atmosphere. Oxidation below the α-β-transformation (1620 F) did not result in embrittlement after an exposure time of 8 hr.
Nitriding	AISI 303 stainless steel	1040	15, 48	0.006	"Malcomizing" patent. Only thin cases can be obtained, since according to Floe (**5**) and Boyer (**21**) the high alloy content apparently slows down the diffusion rate of nitrogen.
Hydrogen Embrittlement	Tough-pitch copper (soft)	1100	0.2 up to 16	0.106	Hydrogen gas at a slightly positive pressure in a tube furnace.
Carburizing	SAE 1020 steel	1650	0.5 up to 24	0.080	Pack-carburized using charcoal.
Hard Chromium Plating	Commercially pure copper (Drawn rod)	140	0.1 up to 4	0.012	Lead anode. A chromium layer exceeding about 0.012 in. chips off easily under tension and thus hardly interferes with the deformation process beyond this thickness.
Induction Hardening	SAE 4340 steel	approx. 1550	...	0.007	Current frequency of 450,000 cps. Stress-relieved at 300 F for one hr after induction-hardening.

Tension specimens were machined from 0.5-in. diameter rod to the dimensions shown in Fig. 1 and then given brittle skins of varying thicknesses as detailed in Table I.[4] Figure 2 gives the ductility of these metals as a function of skin depth.[5]

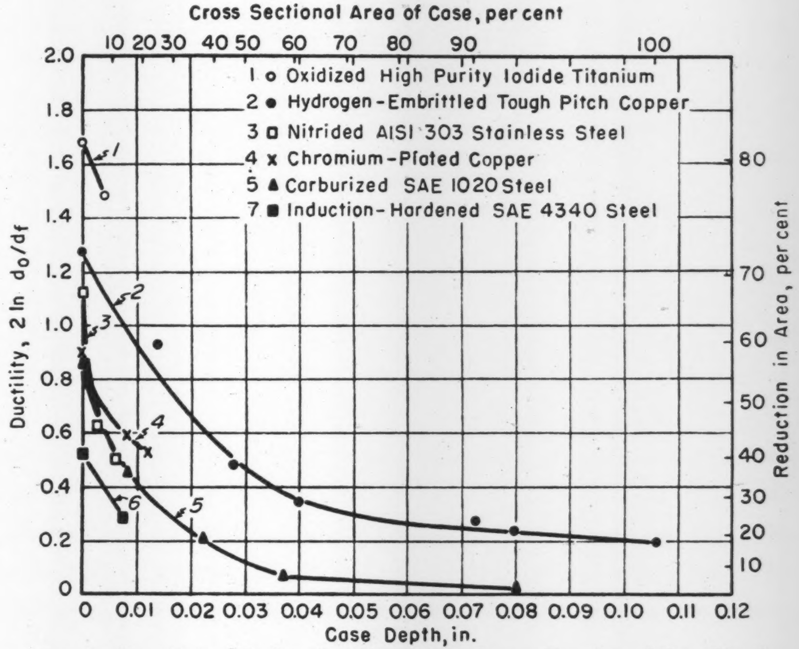

FIG. 2.—Ductility of Surface-Embrittled Metals as a Function of Case Depth.

The ductility drops rapidly at first but falls off more slowly at greater case thicknesses. Percentagewise, the loss in ductility arising solely from a geometric effect would be linearly proportional to the bulk which the skin occupies, assuming that the skin is completely brittle. However, a plot of the ratio of the ductility with a case ϵ_s to that without a case ϵ_o as a function of the percentage of embrittled cross-sectional area, Fig. 4[6] shows that the curve for each metal drops faster than a 45 deg line, indicating that each metal is embrittled to a greater extent than to be expected from a pure geometric effect. The departure from a 45 deg line is most pronounced with the nitrided steel.

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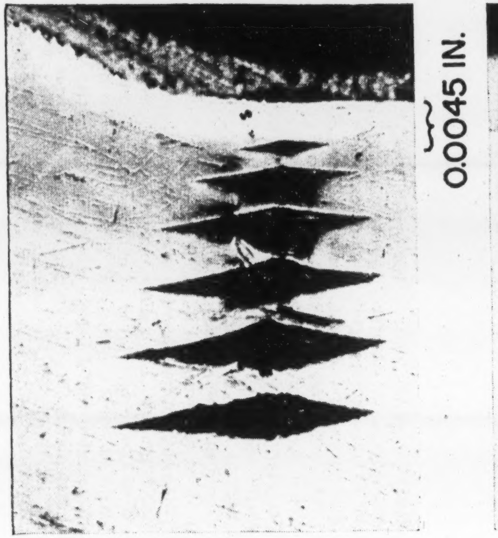
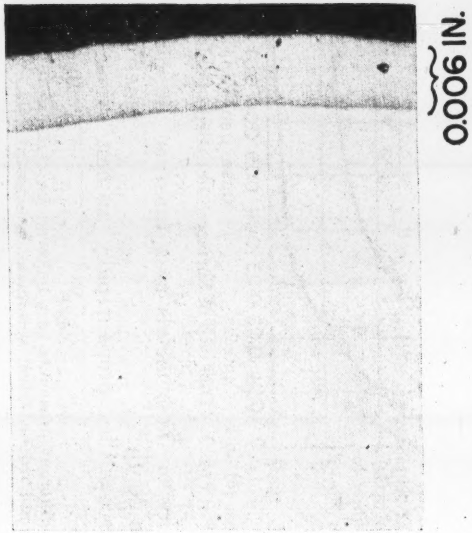
[4] A different procedure was followed for the induction-hardened steel specimens because of the difficulty in obtaining a standard type specimen with a uniform, thin hard case. The corresponding specimens were machined from 0.300 in. diam rods which were already hardened.

[5] Thickness measurements of the brittle surface layers (average of four readings) were made on polished cross-sections, using the micrometer stage of a microscope. In the case of the carburized SAE 1020 steel and the hydrogen-embrittled copper, the brittle skin did not have a sharp boundary (see Figs. 3(*d*) and 3(*f*)). The thickness of these skins was measured on an etched section with a Filar eyepiece under a small magnification only, so that the embrittled area appeared to be a solid band. This method yielded thickness values that fell somewhere in the diffuse boundary. The values indicating the thickness of the surface layer of oxidized titanium pertain to the thickness of the oxygen-nitrogen-saturated metal region only, since the external scale flakes off easily during testing and does not interfere with the deformation behavior.

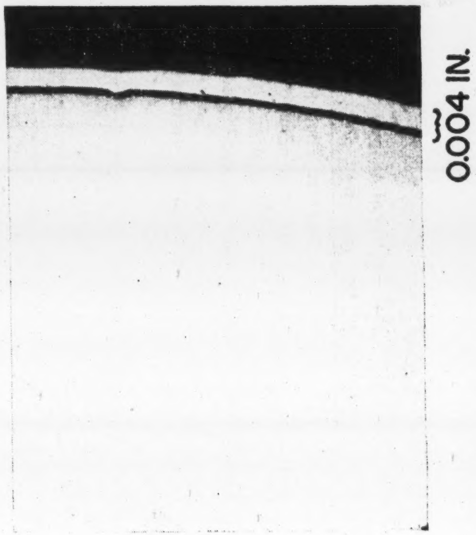
[6] The fact that the skin of hydrogen-embrittled copper displayed some ductility was taken into consideration by subtracting the ductility value of the case alone (taken at total penetration of hydrogen from Fig. 2 and equal to 0.2) from both ϵ_s and ϵ_o, so that the ordinate values in Fig. 4 for copper represent the ratio $\frac{\epsilon_s - 0.2}{\epsilon_o - 0.2}$.



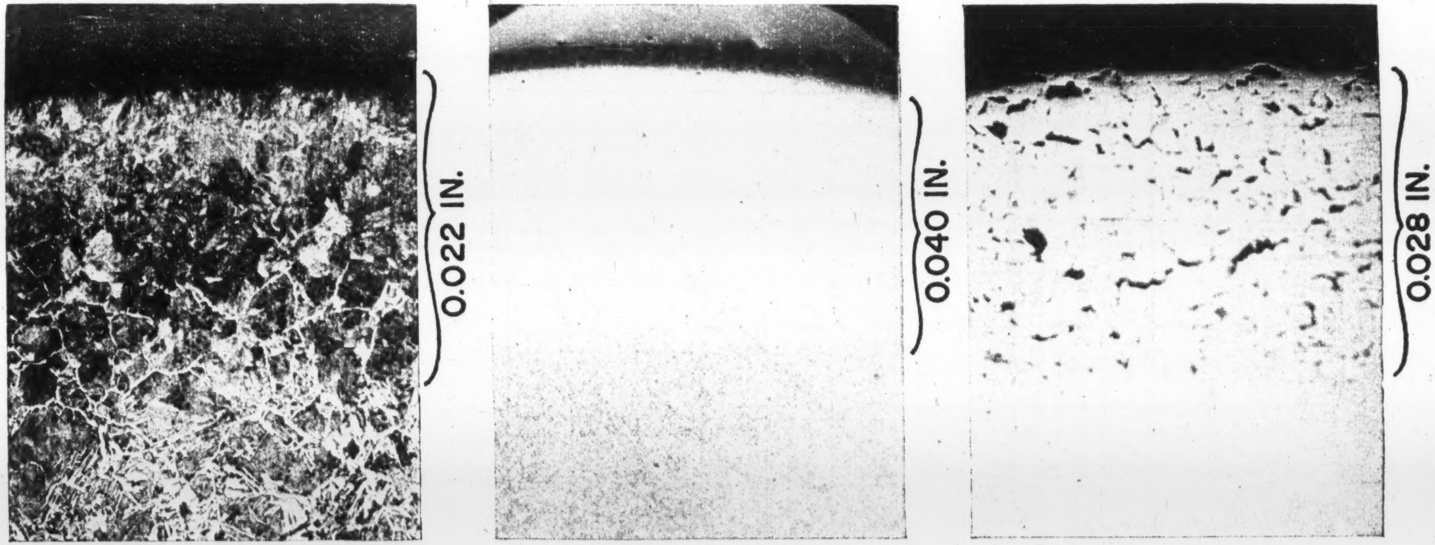
(*a*) Oxidized high-purity iodide titanium (× 75).



(*b*) Nitrided AISI 303 stainless steel (× 60).



(*c*) Chromium-plated copper (× 30).



(*d*) Pack-carburized SAE 1020 steel (× 75).

(*e*) Induction-hardened SAE 4340 steel (× 30).

(*f*) Hydrogen-embrittled tough-pitch copper (× 60).

Fig. 3.—Brittle Skins on the Six Ductile Base Metals Under Investigation (Case Depths as Indicated).

Experimental Series B:

A second series of experiments was run to establish the trend of the ductility recovery upon gradual removal of the skin and to determine whether the embrittlement of the specimens as a whole was due to the presence of the skin alone or not.

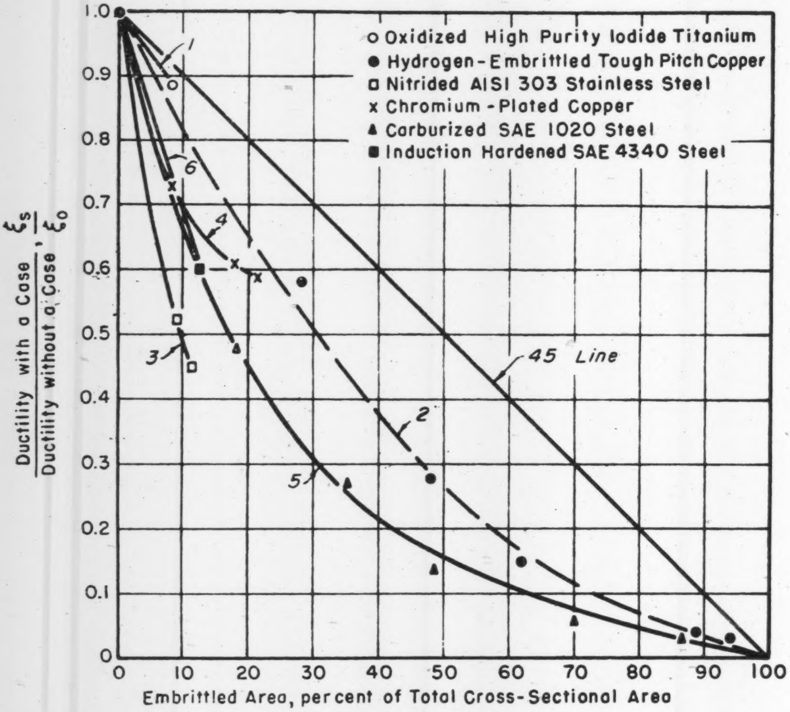


FIG. 4.—Ductility Ratio of Embrittled to Non-Embrittled Specimens as a Function of Per cent Bulk Occupied by the Skin.

About ten specimens of each material were embrittled to the same case depth (which, however, varied from material to material). Before testing, a portion of the original layer was removed by grinding. The test results, Fig. 5, illustrate how the ductility was increased by removing surface layers from the embrittled skin, reaching the ductility level of the base metal (compare with Fig. 2) when the entire embrittled case was ground off. This indicates that the brittle skin is solely responsible for the loss in ductility and that the core did not suffer any permanent damage during the surface-embrittling treatment.

It is instructive to inquire whether the ductility of a specimen with a given case depth reached by exposure to an embrittling atmosphere is the same as that with the same case depth reached by grinding off part of the skin of a thicker case. A comparison of these values for hydrogen-embrittled copper, carburized steel, and nitrided stainless steel Fig. 6 suggests that the ductility is the same for specimens with an "as-embrittled" surface and an "as-embrittled - and - ground" surface.[8] (This comparison was not made for the other three materials studied due to the limited data.)

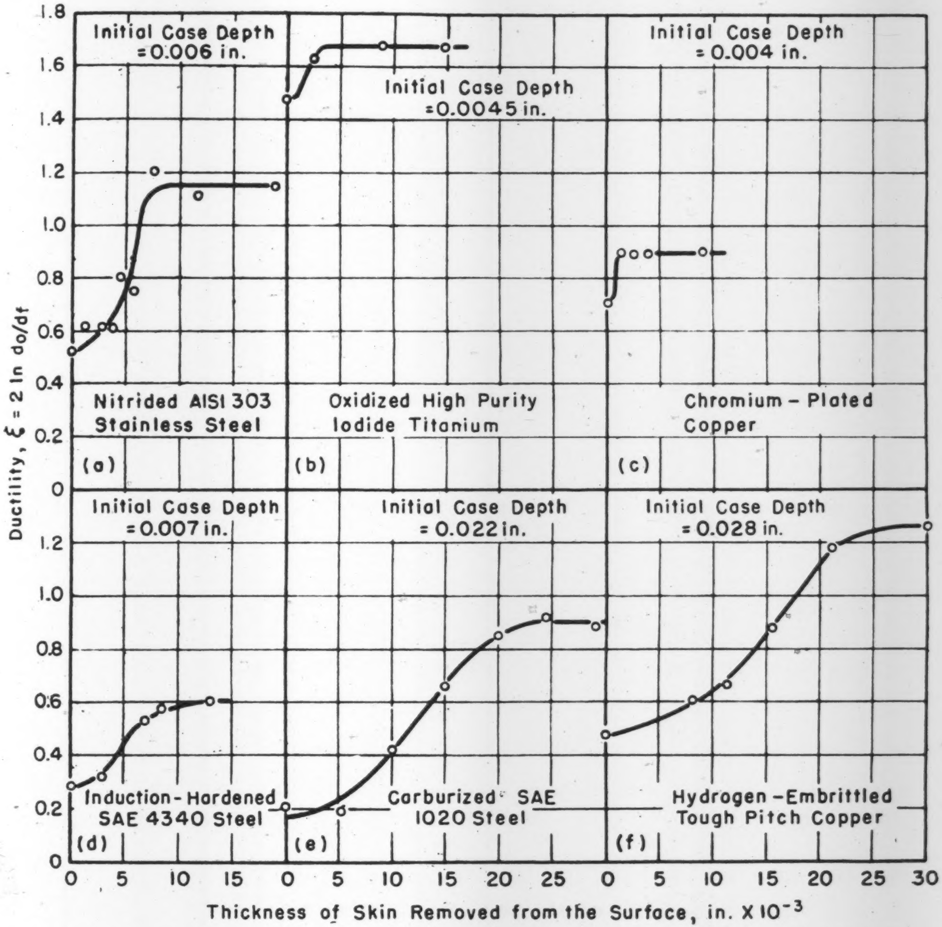


FIG. 5.—Ductility Recovery by Removing Surface Layers from Surface-Embrittled Metals.

siderably higher than that of the underlying metal, with the exception of hydrogen-embrittled copper which showed little, if any, difference in hardness from case to core. From Figs. 3 and 7 it is seen that a sharp change in hardness defines a sharp boundary between case and core, while a gradual change in hardness defines a gradual transition from case to core. Similarly, the hardness results can be related qualitatively to the ductility recovery evidenced in Fig. 5; that is, upon removal of the skin,

Microhardness tests of one specimen of each embrittled metal, Fig. 7, showed that the hardness of the case was con-

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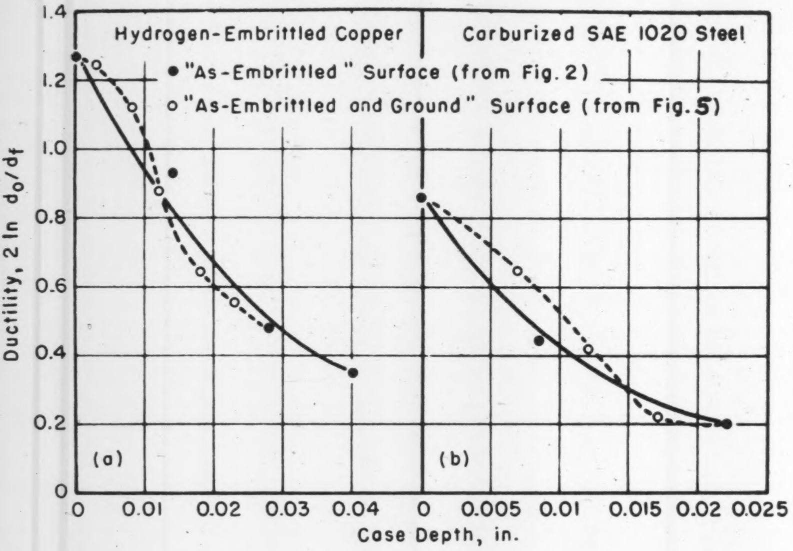
[8] The slight effect of surface condition indicated for thin case depths of hydrogen-embrittled copper and carburized steel may be due to the uncertainty in determining the thicknesses of the embrittled skins since both of these embrittling processes do not give a sharp demarcation between case and core.



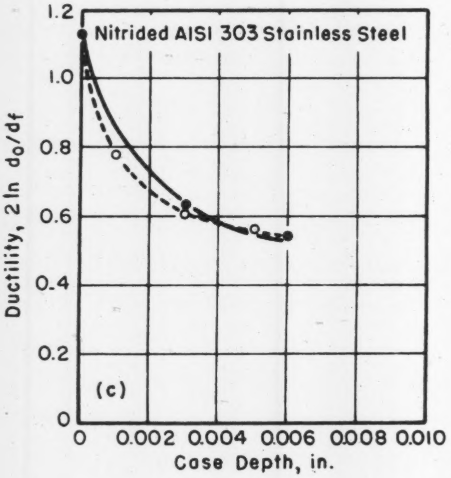


FIG. 6.—Comparison of the Effect of Surface Condition on the Ductility of Various Metals with Brittle Skins.

a steep increase in ductility occurs when the hardness drops sharply at the case-core boundary, while a gradual rise in ductility is associated with a gradual decrease in hardness from case to core. (An exception is hydrogen-embrittled copper.) It is important to note that the ratio of case to core hardness is no measure of the degree of embrittlement to be expected.

Experimental Series C:

Scattered micrographs of interrupted tension tests (Fig. 8) show that, in contrast with the fracturing of a ductile round bar (16–18), cracking of a surface-embrittled specimen starts in the skin and propagates inwards toward the core. These cracks appear to stop near the boundary between case and core and penetrate farther into the core only at a late stage of the tension test. This is suggested by tests in which specimens with brittle skins were strained up to about one-half of their ductility. Before these specimens were subsequently pulled to fracture, various amounts of the case were ground off. As Fig. 9 shows, once the skin was totally removed, the ductility of the core was equal to that of the virgin stock less the prestrain. If the cracks had extended into the base metal the ductility of the core would have been less than this value.

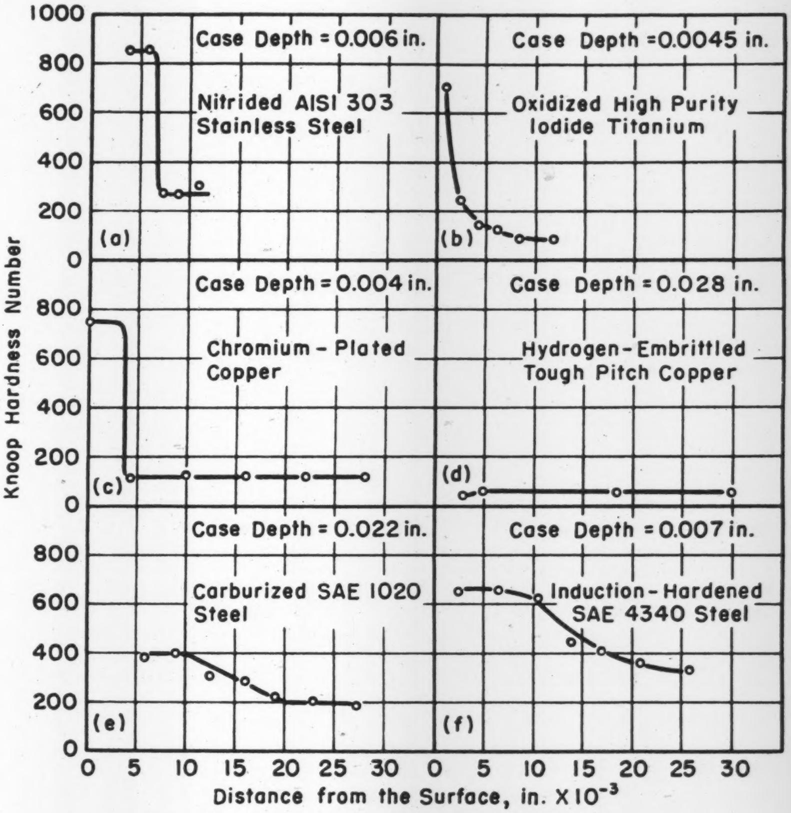
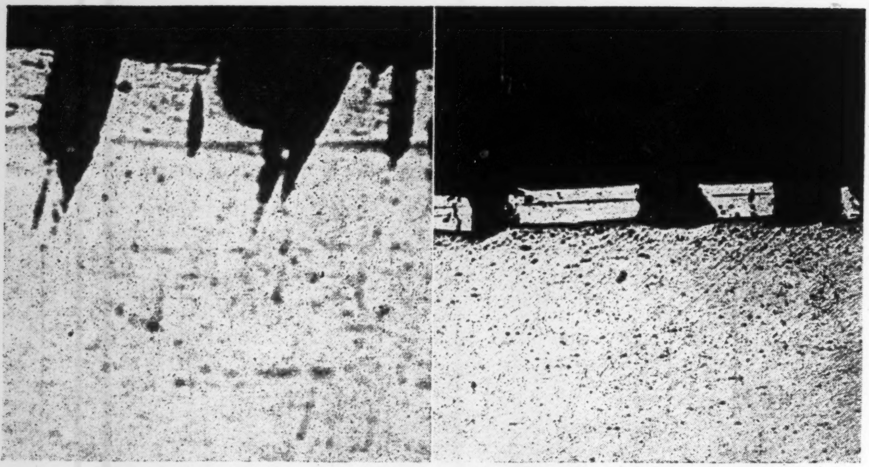


FIG. 7.—Distribution of Microhardness of Surface-Embrittled Metals.

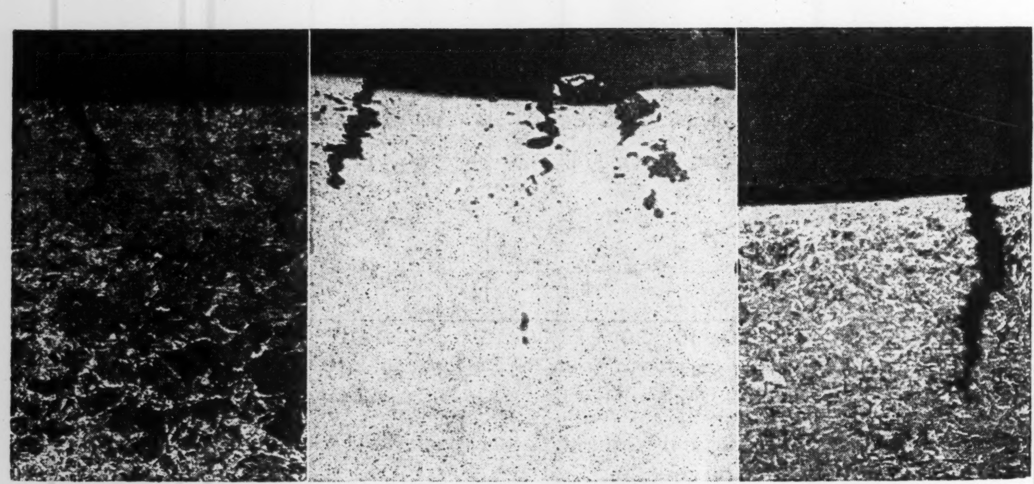
Experimental Series D:

The above results, if inconclusive, at least suggest that the effect of a brittle skin on the over-all ductility of a metal part can be rationalized on the basis that the skin cracks early upon stressing and from that point on the test becomes a notch tension test of the underlying



(*a*) Nitrided AISI 303 stainless steel (× 75).

(*b*) Chromium-plated copper (×50).



(*c*) Pack-carburized SAE 1020 steel (× 75).

d) Hydrogen-embrittled tough-pitch copper (× 30).

(*e*) Induction-hardened SAE 4340 steel (× 50).

FIG. 8.—Cracks Appearing upon Straining of Surface-Embrittled Metals.

core, the depth of the notch being roughly that of the brittle skin.[9]

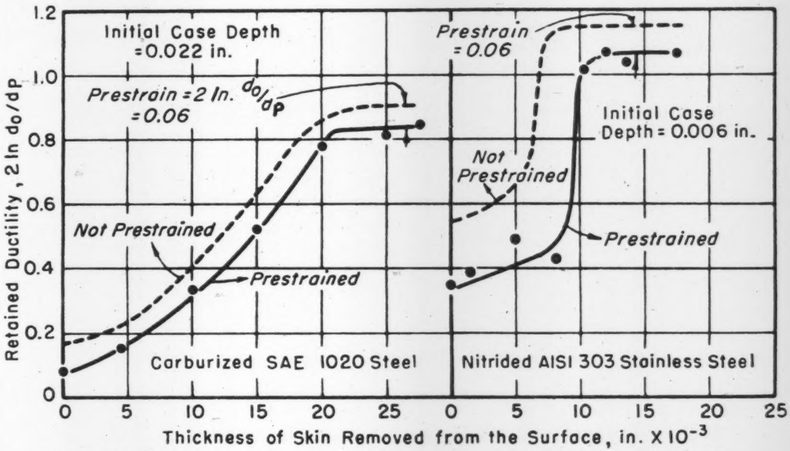


Fig. 9.—Retained Ductility of Prestrained Specimens as a Function of Skin Removed from Surface-Embrittled Metals.
— Retained ductility; - - - Total ductility replotted from Fig. 5.

To check this possibility, a series of notch tension tests (see Fig. 10 for dimensions of test specimens) was run on the SAE 1020 steel in the uncarburized condition. The ductility of this steel with different notch depths is given in Fig. 11(a) together with the ductility of the same steel with different case carburization depths. The curves coincide amazingly well.[10] Furthermore, the data for hydrogen-embrittled copper agree with Ripling's data (19) for the notch tensile ductility of copper (see Fig. 11(b)).

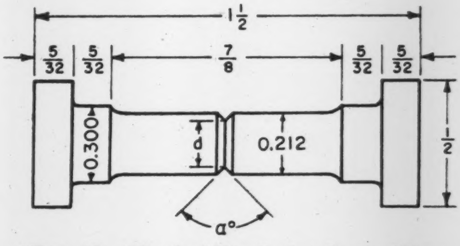


Fig. 10.—Notched Tensile Specimen of Varying Notch Depth and Notch Angle Used in the Study of the Notch-Sensitivity of SAE 1020 Steel (Notch Radius Less Than 0.001 in.)

This evidence strongly favors the

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[9] In a microsection of the skin of hydrogen-embrittled copper such as Fig. 3(f) the voids appear to be discontinuous. It is possible that they are connected giving cracks clear through the skin. Cracking of the skin "early upon stressing" might be interpreted as "before stressing" in this instance. On the other hand, these voids do not appear to be as fully developed as the cracks developed after some stressing (see Fig. 8(d) where the cracks now are quite similar in nature to those found in the other skins. To form cracks running *completely* through the hydrogen-embrittled skin, some stressing is necessary as was the case, for example, in those specimens that were completely penetrated with hydrogen-embrittlement but which still displayed a ductility of about 0.2 before breaking (see Fig. 2).

[10] It would hardly be expected that the curves would coincide exactly. The angle and sharpness of the crack formed in the carburized core is not known exactly, nor is the boundary between the carburized case and the core sharply defined. Moreover the ductilities are calculated in a slightly different manner. The diameters at the root of the notch before and after fracture are conventionally used in the notch tensile test in the formula $\epsilon = 2 \ln d_o/d_f$. The diameters before and after fracture outside the skin are used in the samples with brittle skins.

idea that to a close approximation the ductility of a metal with a brittle skin is given directly by the ductility obtained in a notch tension test with a sharp notch of the same depth as the skin.

Moreover, this correlation would explain why the performance of case-carburized parts is so dependent upon the core. If the core is notch sensitive it should not be used in case-carburized applications.

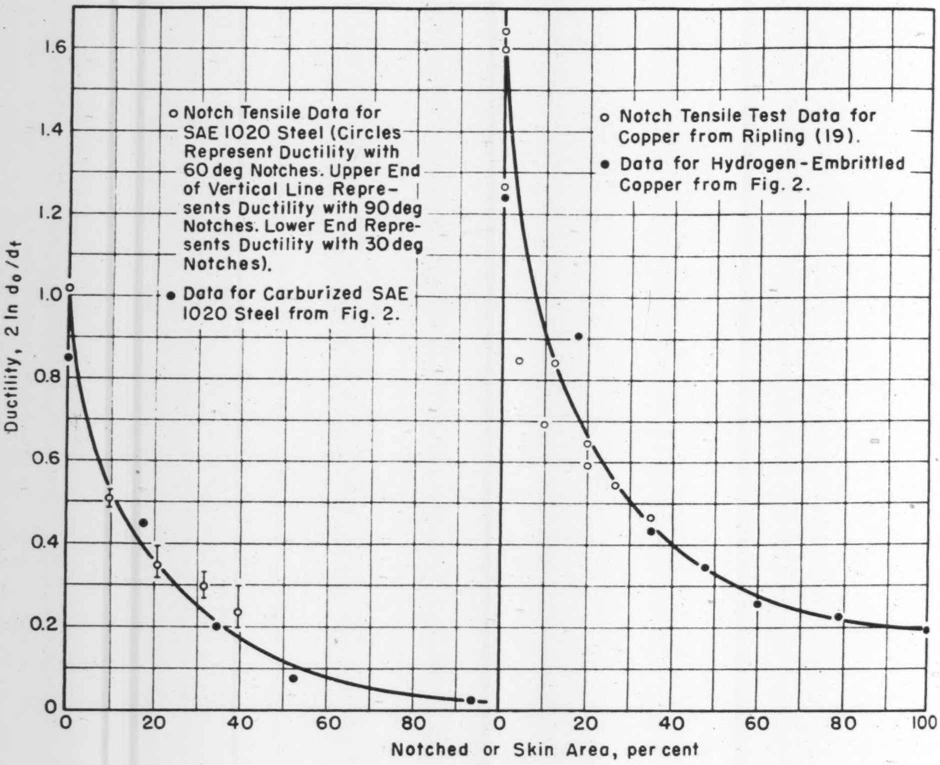

FIG. 11.—Effect of Notched or Brittle Skin Area on Ductility.

This immediately explains why a brittle skin drops the ductility of a metal more rapidly than would be expected from consideration of the area occupied by the skin only, for the notch ductility *versus* notch depth curves for metals in general follow such a path (see Fig. 11 for SAE 1020 steel and copper, Ripling (20) for SAE 1340 steel, Ripling (19) for face-centered cubic metals, etc).

Experimental Series E:

To test this prognostication, the ductility of a highly notch-sensitive material was determined after carburizing to various case depths. An SAE 1340 steel tempered at 600 F was chosen. Ripling (20) has shown that the ductility of this material is sharply lowered by very shallow notches, because they raise the transition temperature to above room-temperature. Fig. 12(*a*) gives the ductility of this steel as a function of carburized case depth. *Although the steel has a fairly good ductility by itself, $\epsilon = 0.65$, a relatively thin case depth drops the ductility of the metal to virtually zero.* This behavior can be contrasted with that of case-carburized SAE 4620—one of the more standard carburizing steels—which, as Fig. 12(*b*) shows, is not affected by the brittle case or by notches any more than chromium-plated copper, for example.

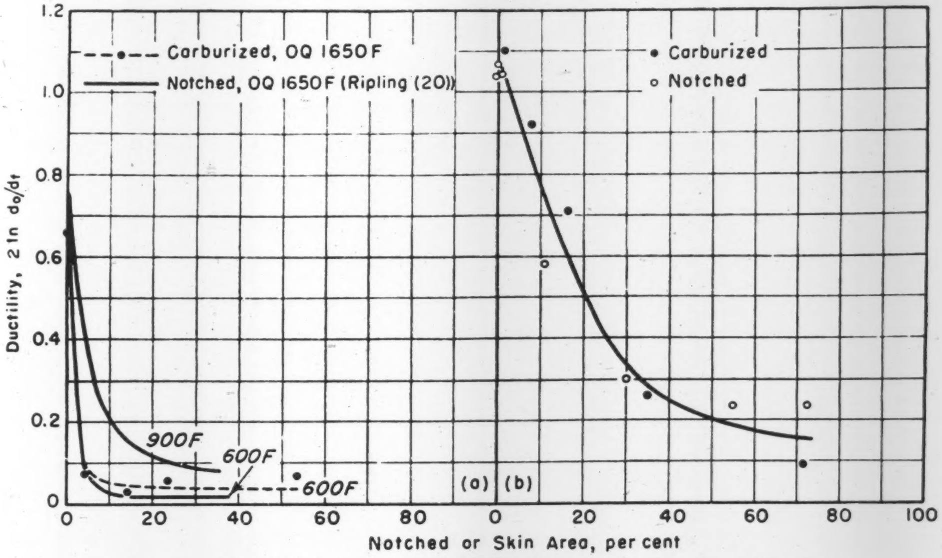

FIG. 12.—Effect of Notched or Brittle Skin Area on the Ductility of Steels.
(*a*) SAE 1340 steel, tempered at indicated temperatures.
(*b*) SAE 4620 steel, furnace cooled from 1650 F.

SUMMARY

The results presented here strongly suggest that a brittle skin on an otherwise ductile material is essentially equivalent to the introduction of a "potential" notch into the metal.

The principal points of evidence for the analogy between the mechanical behavior of a specimen with a brittle skin and that of a notched specimen are:

1. The skin of each of the surface embrittled metals under investigation cracked early in the tension test, thus introducing a circumferential notch into the specimen. The extension of the initially formed crack into the core did not take place until at a late stage of the test.

2. The ductility behavior of a metal under the influence of a varying case depth followed virtually the same pattern as that of the same metal (but without a brittle skin) under the influence of a varying notch depth. This analogy persisted with notch sensitive as well as notch insensitive metals.

It should be borne in mind, of course, that the simple interpretation suggested here of the effect of a brittle surface layer on a ductile core in terms of the notch tensile behavior of the basis material alone should be tempered by other considerations. Some electro-plates may be brittle enough, for example, to crack early in a tension test, but they may also have no adherence whatsoever and flake off before introducing a surface notch into the core (see Table I, for example);[11] residual stresses induced by the surface embrittling procedure (5,21–25) may place a different stress state around the nose of a crack induced by stretching a surface-embrittled metal than that which exists at the base of a machined notch, etc.

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[11] Shedding of the skin could be anticipated on the basis that the ductile core would "pull away" from the brittle skin during the lateral contraction of the specimen on tensile straining. That shedding was an exception rather than the rule is a fact and speculation must yield to it. Possible reasons why shedding was not encountered may be due to such facts as the following: the brittle skin in following the ductile core radially inward would develop compressive hoop stresses (which are less conducive to cracking than the axial *tensile* stresses) and tensile radial stresses but these will be much smaller than the axial tensile stresses (especially for thin skins) by reasons of geometry alone.

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DISCUSSION

MESSRS R. E. RICKSECKER[1] AND T. S. HOWALD[1] (*presented in written form*).—The work carried out in this paper was of real interest to us in the copper industry from two aspects: (1) the quantitative evaluation of the effect of hydrogen embrittlement on loss of ductility, and (2) the profound effect of chromium-plated skins on subsequent ductility of the base metal.

To define further the effect of varying degrees of embrittlement upon the ductility of tough pitch copper strip, we submitted samples of annealed tough pitch copper strip to the authors who undertook the controlled embrittlement of them by heating in hydrogen at 1100 F for varying lengths of time from 5 to 240 min.

Ductility measurements were made by means of an Erichsen cup tester and cup depth at fracture is plotted as a function of depth of embrittlement as shown in the accompanying Fig. 13.

The loss of ductility was most pronounced for the thinnest gage (0.009 in.) tested. It is interesting to note that in those cases where the strips (0.009 and 0.032 in.) were completely embrittled that the ductility did not drop to zero, indicating that some ductility is still available in the embrittled section. A sample of strip heated in air at 1100 F for 240 min had the same cup depth value as the original material, showing that changes in grain size had no effect on the ductility of the copper, and thereby indicated that the ductility loss reported above is attributed to embrittlement.

The loss in ductility for even a very shallow embrittlement is sufficient in many cases to affect seriously the commercial forming of copper strip in gages below 0.010 in.

With regard to the effect of plated skins, we had an interesting customer problem concerning the cracking during forming of chromium-plated copper articles. These parts were drawn from copper strip, chromium plated, and then further formed, at which point some cracking occurred. The cracking was of a variable nature, some parts failed and some did not. Examination of several lots of specimens indicated that drawn, but not chromium-plated parts, would take further forming, thus indicating some effect due to the plating.

Micro-examination indicated that those parts that failed had a chromium-plated thickness of about 0.001 in. while the satisfactory chromium-plated and formed parts had a plating thickness of about 0.0003 in. The real effect of this plate thickness was critically demonstrated by taking chromium-plated parts from the batch that consistently failed (and had a plate thickness of 0.001 in.) and reducing the plate by controlled dissolving of the chromium in an HCl bath. When the plate was reduced to about 0.0003 in. by this method, the part would then successfully form.

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[1] Metallurgical Director, Mechanical Metallurgist, respectively, Chase Brass & Copper Co., Inc., Midwestern Div., Euclid, Ohio.

Figure 14 shows the effect of such chromium-plated skins on the limits of expansion of hard temper copper tube when expanded on a 60-deg conical pin. A plate thickness of 0.001 in. is sufficient to cause a loss of about 43 per cent in ductility, which is quite significant in many forming operations.

These commercial findings are in excellent agreement with the basic findings of this paper and should be given serious consideration in design and fabrication of commercial components.

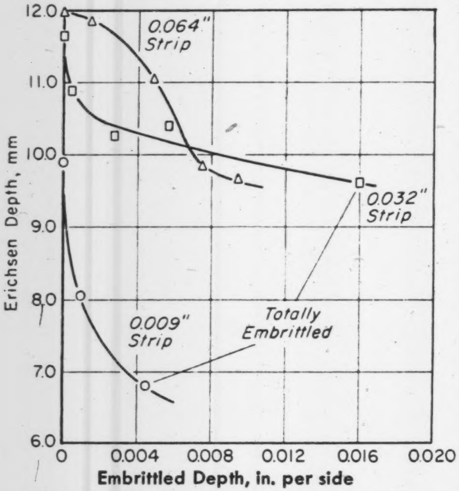

FIG. 13.—Effect of Hydrogen Embrittlement on Ductility of Copper Strip.

MR. O. O. MILLER.[2]—This paper may be of a good deal of interest to people using carburizing steels. For many years the manufacturers of roller bearings and gears and such things have recognized that with carburizing steels there was a tremendous difference between the different types that showed up in very severe uses, such as truck gears, heavy roller bearings, and things like that.

A suitable laboratory test has not been forthcoming to date to duplicate such service tests. Possibly this offers a good practical method to do it. Further work will be needed, in that direction.

MESSRS W. G. FORM AND W. M. BALDWIN, JR. (*authors' closure*).—The findings submitted by Messrs. Ricksecker and Howald supplement very nicely the data presented in our paper, in that they give further evidence that the pronounced loss in ductility due to a very thin brittle layer is not confined to ordinary tension tests only. It would be most informative to carry out Erichsen tests on the same material (annealed tough pitch copper), but on samples provided with an appropriate notch instead of an embrittled layer. Thus the main point stressed in our paper, namely, the apparent analogy between the mechanical behavior of a specimen with a brittle skin and that with a notch could be checked under more severe stress conditions than was done by us.

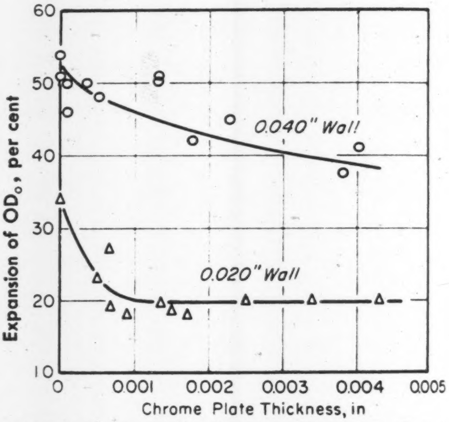

FIG. 14—Expansion Limits of Chrome Plated Phosphorus Deoxidized Copper Tube.

A point of particular interest is the influence of the gage thickness—shown in Fig. 13 of the discussion—to the effect that under a given embrittling condition the loss in ductility increases with decreasing strip thickness. A replot of these data in terms of percentage loss in ductility *versus* percentage embrittled area somewhat reduces the apparently strong dependency upon strip thickness but pronounced differences in degree of embrittlement between the various thicknesses persist.

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[2] International Nickel Co., Research Laboratory, Bayonne, N. J.

We have also carried out some tension tests on geometrically similar specimens of nitrided stainless steel in order to study the effect of specimen size on the degree of embrittlement. However, in the range we examined no size effect was observed. It is well known that the Erichsen test is very gage sensitive, in that the height of the cup formed decreases as the gage thickness is reduced. It is fair to assume that the increased brittleness of thin gages has its adverse effects on the notch behavior of the material, what might account for the sharper drop in ductility of plated or hydrogen-embrittled thin specimens, than this is the case for thicker ones.

The authors wish to express their appreciation to Messrs. Ricksecker and Howald for their interesting contribution and to Mr. Miller for emphasizing the industrial interest in this paper.

NOTCH TENSILE BEHAVIOR OF FACE CENTERED CUBIC METALS*

BY E. J. RIPLING[1]

SYNOPSIS

The notch insensitivity of face-centered cubic metals when tested at a constant rate and testing temperature was found to be a linear function of the unnotched strain hardening exponent.

What are the inherent characteristics of a material that determine its behavior in the presence of notches? In the answer to this question lies the possibility of designing materials that are insensitive to many adverse loading conditions, as well as the possibility of predicting notch performance without the necessity of notch testing.

One of the most perplexing aspects of deducing notch behaviors from unnotched properties results from the fact that similar materials are not equally harmed by the introduction of notches. Many materials whose engineering tensile properties are almost identical perform quite differently in the presence of notches. Yet the component features that differentiate a test using a smooth bar (from which engineering data are collected) from one employing a notched test section, that is, the development of a stress concentration and transverse tensile stresses, are completely defined (at least before the specimen is strained) by the geometry of the test piece and are independent of the material being tested. Of these two embrittling conditions that are developed on the introduction of a notch, stress concentration and triaxiality, only the latter remains effective after the test specimen has undergone a slight amount of plastic flow. According to Sachs and Lubahn (1)[2] an average strain of about two per cent overcomes the stress concentration, so that the presence or absence of transverse stresses is the only difference between notched and unnotched bars after small deformations.

The notch performance of any product whose notch ductility is in excess of a few per cent then should be deducible from its unnotched behavior by considering only the action of a transverse tensile stress superimposed upon the applied longitudinal stress. The notch ductility of a metal may be either slightly or greatly in excess of a few per cent depending on its ability to deform in the presence of triaxial tensile stresses or else depending on its ability to attenuate the transverse stresses on straining.

Transverse stresses are developed in a

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] Formerly Department of Metallurgical Engineering, Case Institute of Technology, Cleveland, Ohio; now with Continental Can Co., Central Research and Engineering Div., Chicago, Ill.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 671.

notched bar of the type shown in Fig. 1(*a*) as a result of the flow restraint that each cross-sectional element imposes on its smaller neighbor. The application of a load, P, to the cylindrical portion of the test specimen develops a stress equal to P/A in the cylindrical part of the bar. The reaction to this stress is, of course, a contraction which is uniform along the length of the cylinder. The application of the load, P, produces a stress larger than P/A in the notched section immediately adjacent to the cylinder because of its reduced cross-section. This larger stress tends to produce a larger unit contraction in each cross-sectional element of the notched portion of the bar than is produced in its larger adjacent neighbor. Since the cross-sectional elements in the notched section must remain compatible, each element in the reduced portion of the bar imposes a transverse tensile stress onto its smaller neighbor in order to restrict this excessive contraction.

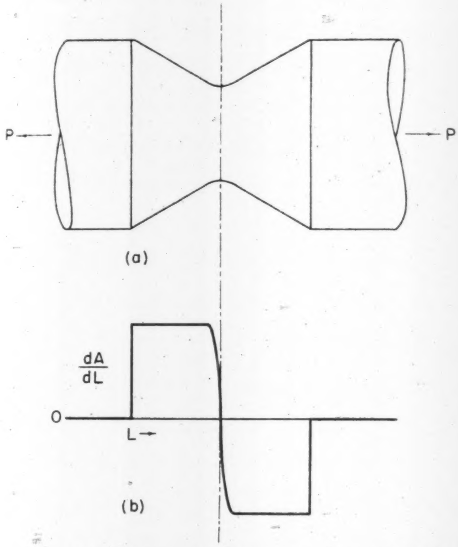

FIG. 1.—Schematic Representation of Notch-Bar Contour and Variation of dA-dL Along Its Length.

Hence, the transverse stresses are seen to develop from the difference in contractile tendency of the various elements along the length of the notched section. If a metal is sufficiently ductile to allow plastic flow of all the material in the vicinity of the notch bottom, the contractile tendency of each element in the test section is dependent on the product of the flow stress and the cross-sectional area, that is, it is dependent on the load necessary to produce plastic flow in each cross-sectional element. Consequently, the triaxiality (ratio of transverse to longitudinal stress) within rather wide limits, is a function of the manner in which the tendency to contract (flow stress × cross-sectional area for plastic strains) changes with position along the specimen axis,

$$T = f\int \left(\frac{\mathrm{d}(k \times A)}{\mathrm{d}L}\right) \mathrm{d}L \ldots\ldots (1a)$$

where:

T = triaxiality,
k = flow stress,
A = cross-sectional area, and
L = axial distance from cylindrical portion of test bar.

TABLE I.

Material	Treatment
Annealed copper....	Annealed at 900 F for 1 hr, air cooled
Annealed nickel......	Annealed at 1400 F for 1 hr, air cooled
Annealed monel.....	Annealed at 1700 F for 1 hr, air cooled
AISI 310 stainless steel.............	Stress relieved at 1500 F for 1 hr, air cooled
24S-T4 aluminum....	Solution heat treated at 920 F for 1 hr, water quenched, aged 4 days at room temperature
75S-T6 aluminum....	Solution heat treated at 900 F for 45 min, water quenched, aged 210 F for 5 hr, 315 F for 10 hr

Equation 1a can be rewritten as,

$$T = \int\!\!\int \left(k \frac{\mathrm{d}A}{\mathrm{d}L} + A \frac{\mathrm{d}k}{\mathrm{d}L} \right) \mathrm{d}L \ldots (1b)$$

So long as k is a constant, the second term in Eq 1b equals zero, and the triaxiality is a function of specimen geometry $\left(\frac{k\mathrm{d}A}{\mathrm{d}L} \right)$ only. The quantity $\mathrm{d}A/\mathrm{d}L$ varies as shown in Fig. 1(b). The integrated value of $\mathrm{d}A/\mathrm{d}L$ is seen to be highly dependent on the notch depth and notch angle, but only slightly dependent on the radius at the notch bottom before straining.[3]

Although $\frac{\mathrm{d}k}{\mathrm{d}L}$ is equal to zero at the start of any test, k increases with plastic strain for all materials that strain harden. Since the strains along the length of the test specimen increase as A decreases, the product of these two terms ($k \times A$) changes at a continuously slower rate along the length, L, of the notched section as strain proceeds; hence the magnitude of triaxiality decreases with strain. Practically all metals strain-harden so that for any real metal, strain continuously attenuates the initial triaxiality that was imposed upon the notched bar by the specimen's geometry. The rate at which the product ($k \times A$) decreases with deformation depends on the metal being strained. The yield strength, k, changes with strain at a rate which is given by the well-known strain hardening exponent, n, in the empirical equation,

$$k = k_0 \epsilon^n \ldots\ldots\ldots\ldots (2)$$

where:

k_0 = flow stress at a strain of $\epsilon = 1.0$, and

ϵ = natural strain, $\ln \frac{A_0}{A}$.

The higher the strain hardening exponent of a material, the greater is the rate at which $\frac{\mathrm{d}(k \times A)}{\mathrm{d}L}$ decreases with strain, and, hence, the greater the rate at which the triaxiality and the embrittlement of the material are eliminated.

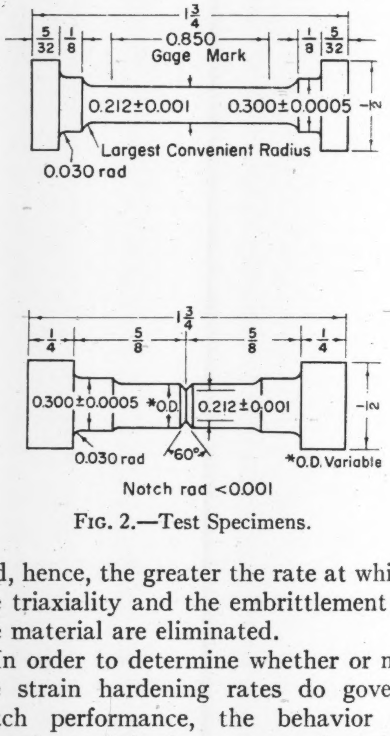

FIG. 2.—Test Specimens.

In order to determine whether or not the strain hardening rates do govern notch performance, the behavior of several metals in the presence of notches was compared with their strain hardening exponents. The notch properties of metals which exhibit a transition temperature are to a large extent dictated by the transition behavior of the metal (**2**, **3**). Consequently, the simpler-behaving face-centered cubic metals were selected for this investigation. Six face-centered cubic metals representing a wide range of strain hardening exponents were tested under varying notched conditions in order to correlate their notch behaviors with their strain hardening exponents.

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[3] This is in agreement with the analysis of Sachs and Lubahn who state that variations in notch radius over a rather wide range do not appreciably change the triaxiality in a notched tensile bar, although they do materially alter the stress concentration factor (**1**). These authors also state that the triaxiality was little changed by permanent strains; but, since their data was all collected on high strength steel, all their strains as well as the strain hardening exponents were quite small.

MATERIAL AND PROCEDURE

The materials used in this investigation were annealed copper, an annealed austenitic stainless steel (AISI 310), annealed nickel, annealed monel, and the heat-treated aluminum base alloys, 24S-T4 and 75S-T6,—all heat treated as shown in Table I. All specimens were completely heat treated before final machining to the dimensions shown in Fig. 2.

Two notched test series were conducted on these materials, one at room temperature and one at −321 F. All tests were performed at a strain rate equivalent to a movement of the test machine head of approximately 0.05 in. per min in a specially designed fixture that produced less than 0.001 in. between the specimen axis and the loading axis (4). Diameter measurements were made

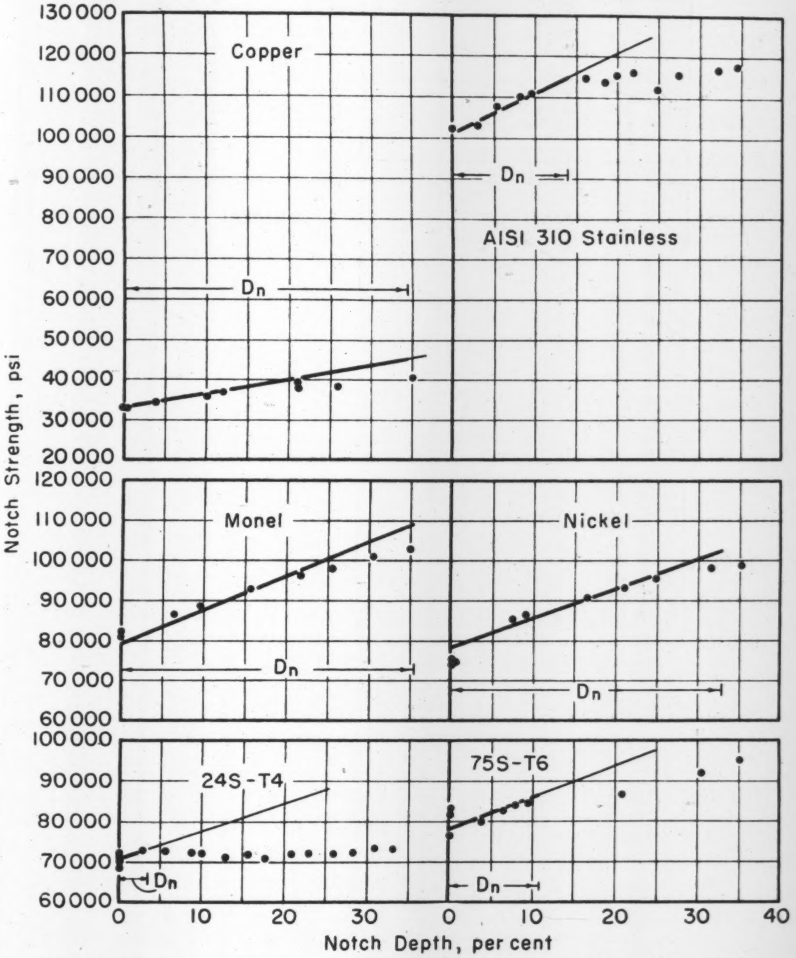

FIG. 3.—Notch Strength as a Function of Notch Depth for a Variety of Face Centered Cubic Metals Tested at Room Temperature.

during the test by means of a specially designed radial strain gage.[4] Notch ductility was calculated from diameter measurements of the notch bottom before and after testing.

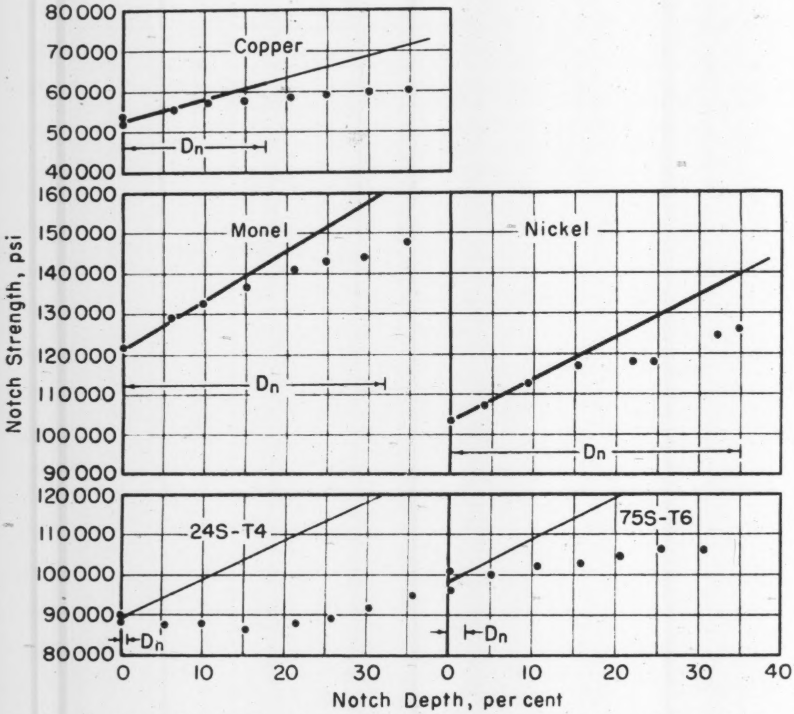

FIG. 4.—Notch Strength as a Function of Notch Depth for a Variety of Face Centered Cub Metals Tested in Liquid Nitrogen (−321 F).

TEST RESULTS AND DISCUSSION

The magnitude of notch severity in a notched tension specimen can be varied by changing either the notch depth, the radius at the bottom of the notch, or the acuity of the notch angle. In order to make specimen preparation as simple as possible, the notch depth was selected as the variable in this investigation. The depths were changed by varying the outside diameter of the test specimens as shown in Fig. 2(*b*).

The effect of notch depth on the notch strength and on the notched ductility for the materials studied is shown in Figs. 3 to 6.

As suggested by Sachs and Lubahn (4), the notch strength rises linearly with increasing notch depth at a rate such that the notch strength is equal to 1.5 times the tensile strength for a 50 per cent notch, for notch specimens whose ductility exceeds the necking strain of the metal. The notch strength then can be described by the following equation so long as the notch ductility exceeds the necking strain:

$$\text{Notch strength} = \text{tensile strength}\;[1 + 0.01(\text{notch depth, per cent})] \quad \ldots (3)$$

Obviously, the notch strength is simply a function of specimen geometry for ductile specimens and is a function of the geometry and ductility when the ductility is low. Straight lines representing Eq 3 as well as the maximum notch depths which produced a notched ductility in excess of the unnotched necking strain, D_n, have been added to the graphs in Figs. 3 and 4. Since the notch strength, in the range of values in which it is of most interest, is a function of the notch ductility, the damage to notch ductility is used as the criterion for notch sensitivity in this investigation.

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[4] To be described in a subsequent report.

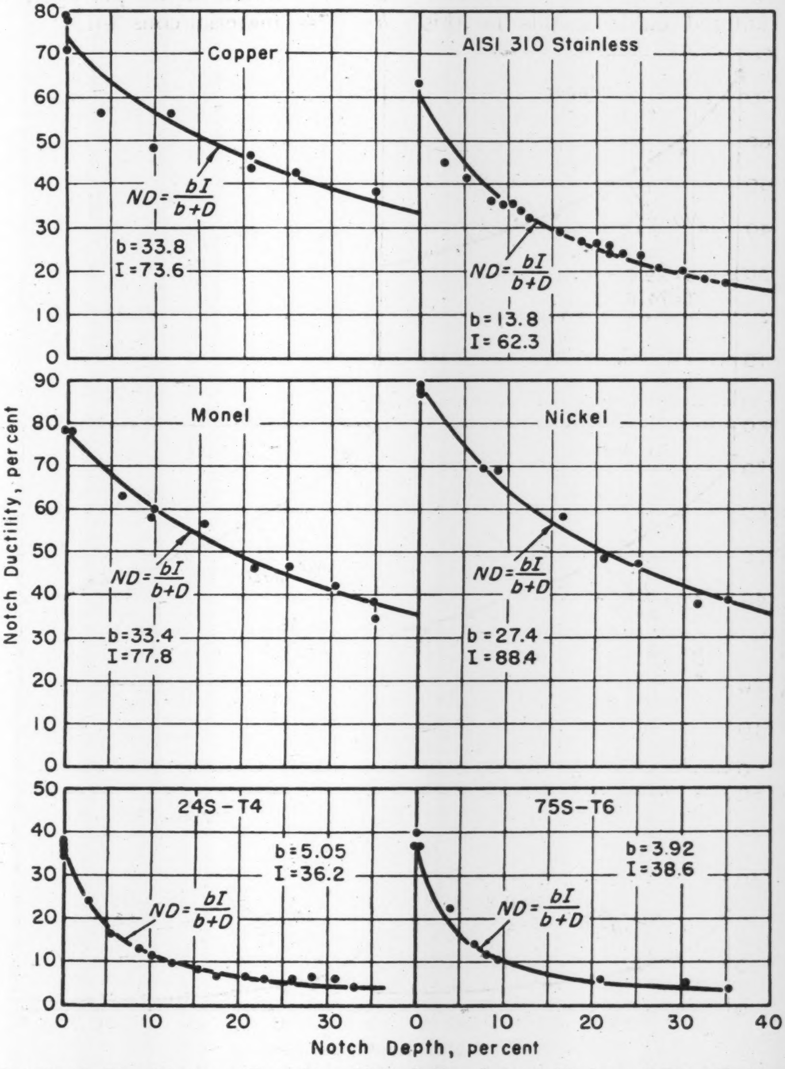

FIG. 5.—Notch Ductility as a Function of Notch Depth for a Variety of Face Centered Cubic Metals Tested at Room Temperature.

Notch sensitivity can be best defined by means of a quantity which measures the harm done to a material's ductility on the introduction of a notch in such a manner that the quantity is not influenced by the absolute value of unnotched ductility. All curves of notch ductility *versus* notch depth, both those obtained on face-centered cubic metals in this investigation, Figs. 5 and 6, as well as those previously presented for body centered cubic (3) and hexagonal (5, 6) metals, are of the form:

$$\text{N.D.} = \frac{bI}{b + D} \qquad (4)$$

where:

N.D. = notch ductility, per cent,
I = unnotched ductility, per cent,
D = notch depth, per cent, and
b = material constant.

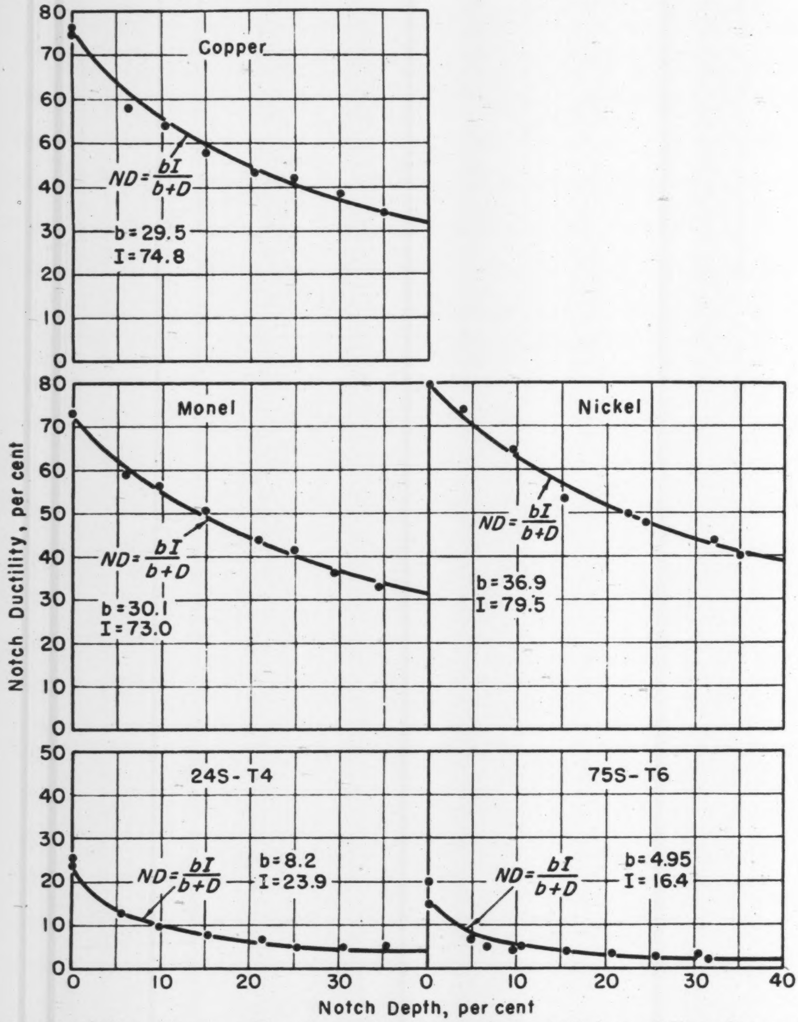

FIG. 6.—Notch Ductility as a Function of Notch Depth for a Variety of Face Centered Cubic Metals Tested in Liquid Nitrogen (−321 F).

Equation 4 represents an hyperbola which asymptotically approaches the abscissa (notch depth) axis and an ordinate axis displaced b units of negative notch depth into the second quadrant as shown schematically in Fig. 7(a). As b decreases, Eq 4 describes a family of hyperbolæ which fall at an increasingly rapid rate in the first quadrant, Fig. 7(*b*). Hence *b* is a measure of notch insensitivity. Large values of *b* represent gradual losses of ductility with increasing notch severity, while small values of *b* represent rapid ductility losses with increased notch severity. If the quantity *z* is introduced to represent the amount of the unnotched ductility that is lost by the addition of a notch of some given shape, as shown schematically in Fig. 7(*a*), the proportion of the unnotched ductility that is sacrificed by notching is given by:

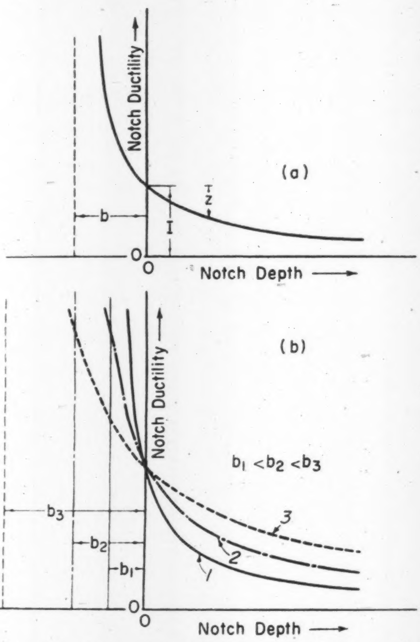

FIG. 7.—Schematic Diagrams Showing the Effect of "*b*" on Loss in Unnotched Ductility.

$$\frac{z}{I} = \frac{I - N.D.}{I} = \frac{D}{b + D} \quad \ldots\ldots(5)$$

The curves in Figs. 5 and 6 were calculated by converting Eq 4 to a linear equation after which the values of *b* and *I* were obtained by the method of least squares. In order to give relatively equal weight to the unnotched and notched ductility values and at the same time to insure that the unnotched ductility points were heavily weighed, the average unnotched ductility was given the same weight as the sum of the notch ductility values. It is seen in Figs. 5 and 6 that the data points do describe hyperbolæ.

Absolute changes in *b*, unfortunately, do not represent absolute changes in notch ductility. A given change in *b*, when the *b* value is large, produces a small change in notch behaviors as shown schematically by the pair of curves whose *b* values differ by 2 ($b = 30$ and $b = 28$) in Fig. 8. This same absolute change in *b* when the *b* value is small, $b = 3$ as compared with $b = 1$, on the other hand, results in large differences in notch performance, Fig. 8. For this reason, notch insensitivity is best defined by introducing the quality b' where:

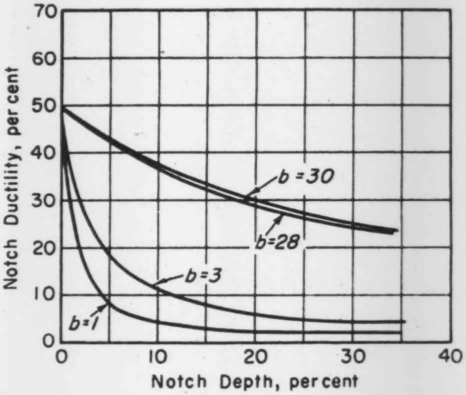

FIG. 8.—Change in Notch Ductility Produced by a Small Change in *b* for a Notch Brittle and a Notch Ductile Material.

$$b' = \log_{10} b \quad \ldots\ldots\ldots\ldots(6)$$

Changes in notch sensitivity on the basis of this criterion are proportional to percentage differences in *b*.

It is on the basis of their influence on b' that strain hardening exponents are related to notch sensitivity in this investigation.

Strain hardening exponents, *n* were obtained in the study by plotting the true stress[5] *versus* the natural strain on log-log paper. No subtractions were made for elastic strains. Instead the effect of elastic strains was minimized and at the same time the necessity of making corrections for necking was avoided by taking the strain hardening exponent as the slope of the straight-line portion of the log k *versus* log ϵ curves at strains just below the maximum load strain.

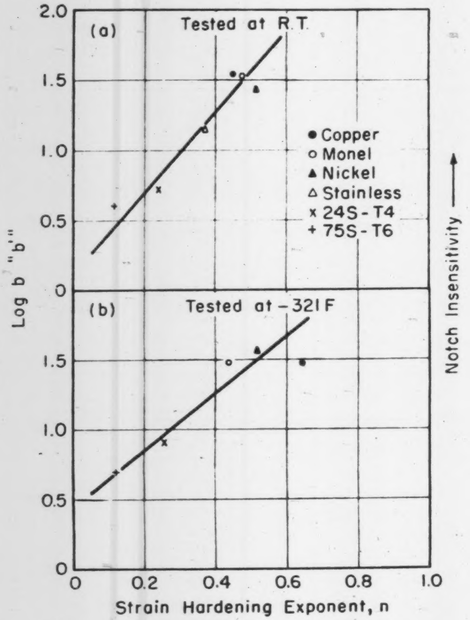

FIG. 9.—Dependence of Notch Insensitivity Log b "b'" on Unnotched Strain Hardening Exponent, n.

The notch insensitivity[6] is seen to be linearly related to b' in Fig. 9(*a*) and (*b*) so that the effect of strain hardening exponents on notch insensitivity is given by the equation:

$$b' = p + qn \quad \ldots\ldots\ldots\ldots(7)$$

where $p = 0.12$ and $q = 2.9$ on testing at room temperature, and $p = 0.042$ and $q = 2.1$ on testing at -321 F.

Since a single equation cannot be used to describe the notch sensitivity at two testing temperatures, notch behavior of face-centered cubic metals is not a unique function of the strain hardening exponent. However at a constant temperature, notch sensitivity is determined by $\frac{dk}{dL}$ as suggested above.

By means of Eq. 4, 5, and 6, the notch ductility can be predicted from data collected on unnotched test specimens (unnotched ductility and strain hardening exponent),

$$\text{N.D.} = \frac{I \text{ antilog } (p + qn)}{\text{antilog } (p + q) + n} \quad \ldots (8)$$

It is interesting to note that the notch insensitivity criterion suggested above also was found to be simply related to other material and testing variables. For example, b' was found to be linearly related to the nitrogen content of a series of titanium-nitrogen alloys, (6). These same alloys, as well as a heat-treated steel were found to show a linear temperature dependence of notch insensitivity over a rather wide range of testing temperatures (6).

Acknowledgments:

The author acknowledges the many helpful discussions he had with Dr. F. J. Anders and Prof. L. J. Ebert during the course of this investigation.

Thanks are also due to R. G. Howe and G. Condzer who conducted the tests.

A debt of gratitude is also due to The International Nickel Co. for supplying the nickel and monel.

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[5] In order to aid in calculations of true stresses in this and other investigations, a true stress calculator was developed that directly converted diameter changes and load readings to true stress values.

[6] Since both b and b' increase as a material becomes less damaged by notching, these quantities measure notch insensitivity rather than the more common notch sensitivity.

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MATERIALS FOR HELICAL COMPRESSION SPRINGS FOR USE AT CONSTANT DEFLECTION FROM 600 TO 1400 F*

By R. G. Matters and R. E. Lochen[1]

Helical compression springs made from the common spring materials will not satisfactorily support even moderate loads at temperatures above about 500 F. At elevated temperatures, the elastic strain in the outer fibers of the spring under load is gradually changed to plastic strain, and permanent set or relaxation of the spring occurs. There is no simple relationship between torsional stress and torsional strain in the spring wire after relaxation has occurred. Hence, relaxation behavior is not amenable to a strict quantitative evaluation, and relaxation tests of spring materials are largely comparative.

Very little has been published on the relaxation behavior of spring materials at elevated temperatures. Zimmerli (**1**)[2] reported relaxation tests of carbon and low-alloy steel springs at temperatures up to 350 F and of 18-8 stainless steel springs at temperatures up to 550 F. In his tests, springs were loaded to a fixed deflection corresponding to a given initial stress, then exposed to elevated temperatures for a fixed length of time, unloaded after exposure, and the free length of the spring was measured. The decrease in load was then calculated from the change in length of the spring. The data were reported as the per cent loss in load *versus* the initial stress for a constant exposure time for any one spring material, 72 hr for carbon and low-alloy steel and 10 days for the 18-8 stainless steels. Betty, MacQueen, and Rolle (**2**) reported similar relaxation tests on Monel springs at temperatures up to 500 F, "K" Monel springs at temperatures up to 600 F, "Z" nickel springs up to 650 F, and Inconel springs up to 700 F. Zimmerli and Wood (**3**) reported extensive data on endurance limits and some data on relaxation of springs at temperatures up to 650 F.

Over the past ten years, in this laboratory, investigations have been made on a number of materials for application as helical springs under constant deflection conditions. For this specific application, the springs may relax but must support a specified minimum dead load.

TORSIONAL STRESS AND STRAIN IN HELICAL SPRINGS UNDER RELAXATION CONDITIONS

The relationships between torsional stress and torsional strain of a spring under relaxation with a fixed deflection are shown diagrammatically in Fig. 1. Relaxation occurs most rapidly in the outer fibers of the wire which were initially most highly stressed. There is a tendency to equalize the stress over the cross-section of the spring wire as relaxation proceeds. If the restraint is removed, the spring will recover partially but will not attain its original length, and a residual stress pattern will be established. The residual stresses near the center of the spring wire will be in the same direction as the load stresses Near the outside of the spring wire, the direction of the residual stresses will be opposite to the load stresses.

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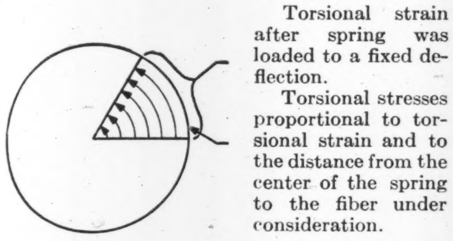
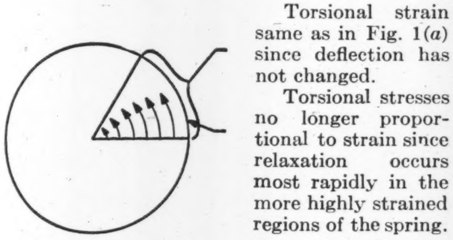
* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] Assistant Director of Research and Supervisor of Physical Metallurgy Section, respectively. Research Laboratories, Allis-Chalmers Manufacturing Co., Milwaukee, Wis.

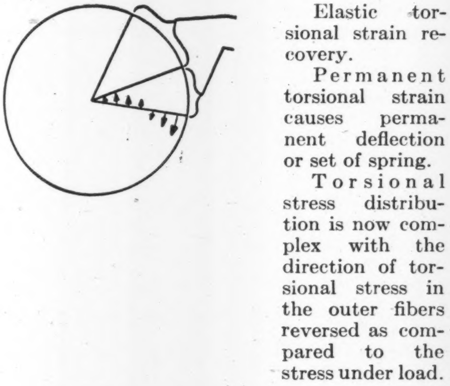
[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 683.


Torsional strain after spring was loaded to a fixed deflection.
Torsional stresses proportional to torsional strain and to the distance from the center of the spring to the fiber under consideration.

(*a*) Stress and strain immediately after spring was loaded.


Torsional strain same as in Fig. 1(*a*) since deflection has not changed.
Torsional stresses no longer proportional to strain since relaxation occurs most rapidly in the more highly strained regions of the spring.

(*b*) Stress and strain after some relaxation has occurred. Spring still loaded to fixed deflection.


Elastic torsional strain recovery.
Permanent torsional strain causes permanent deflection or set of spring.
Torsional stress distribution is now complex with the direction of torsional stress in the outer fibers reversed as compared to the stress under load.

(*c*) Torsional stress and strain after spring is unloaded.

FIG. 1.—Diagrammatic Representation of Torsional Stress and Strain in Helical Spring Wire Under Relaxation Conditions.

The torsional elastic recovery that occurs when the spring is released is a measure of its load-carrying capacity at the given deflection after relaxation has occurred. For convenience, this torsional elastic recovery can be converted by the usual spring formulas to an effective residual stress in order to compare different spring materials. The results in this paper have been reported on the basis of this effective residual stress, although it is fully recognized that this is a fictitious stress which does not exist in the spring if relaxation occurs.

MATERIALS

At the time these investigations were begun, high-speed steel in the quenched-and-tempered condition was considered to be one of the best available materials for service above 600 F. However, at temperatures approaching 900 F, excessive relaxation of the high-speed steel springs occurred during service. The first materials investigated and compared with high-speed steel included annealed Inconel, cold-drawn type 302 stainless steel, and cold-drawn Timken 16-25-6. More recently, another cold-drawn austenitic material, 19-9-DX, has been investigated. As operating temperatures increased, other materials which may be classed as precipitation hardening "super alloys" were also investigated. The alloys of this type investigated included S-816, Elgiloy, Inconel X, and A-286. The nominal chemical compositions of the materials investigated are given in Table I.

The relaxation behavior of springs made to the following designs were determined:

	Type A	Type B
Free height, in.	1.000 ±0.005	0.938 ±0.005
Solid height, in.	0.625 ±1/64	0.688 ±1/64
Wire diameter, in.	0.063	0.121
Outside diameter, in.	0.500	1.250
Active coils	8	3¼
Ends	Closed and ground square with axis	

Springs of type A design were used for most of the relaxation tests, and only a few tests were made on the larger type B springs. In general, the springs were cold formed by a commercial spring vendor and were subsequently treated in the laboratory as recommended by the supplier of the wire. In some cases, the wire supplier made no recommendation as to the heat treatment of the spring

TABLE I.—NOMINAL CHEMICAL ANALYSES OF SPRING MATERIALS.

	High-Speed Steel	Type 302	Inconel	Timken 16-25-6	19-9-DX	Elgiloy	Inconel X	A-286	S-816
Carbon	0.70	0.10	0.05	0.08	0.30	0.15	0.04	0.08	0.40
Manganese	0.30	1.50	...	...	...	2.00	...	...	...
Silicon	0.30	0.50	...	...	...	...	...	...	...
Chromium	4.00	18.00	14.50	16.50	19.25	20.00	15.00	14.75	20.00
Nickel	...	8.00	78.00	25.00	9.00	15.00	73.00	25.00	20.00
Molybdenum	...	...	...	6.25	1.50	7.00	...	1.25	4.00
Titanium	...	...	...	...	0.60	...	2.40	1.90	...
Tungsten	18.00	...	...	...	1.25	...	...	...	4.00
Vanadium	1.00	...	...	...	...	...	...	0.25	...
Aluminum	...	...	...	...	...	...	0.60	0.35	...
Cobalt	...	...	...	...	...	40.00	...	...	44.00
Columbium	...	...	...	...	...	...	1.00	...	4.00
Nitrogen	...	...	...	0.15	...	...	...	...	...
Beryllium	...	...	...	...	...	0.04	...	...	...
Iron	Bal.	Bal.	7.00	Bal.	Bal.	Bal.	7.00	Bal.	4.00

TABLE II.—SPRING TEST INFORMATION.

Material	Spring Scale, lb per in.		Torsional Modulus, million psi					Condition of Materials
	Type A	Type B	600 F	800 F	1000 F	1200 F	1400 F	
High Speed Steel	32–34	...	...	...	...	...	...	Cold drawn, coiled
Type 302	32	...	...	...	...	...	...	Cold drawn, coiled
Inconel	31–32	...	...	...	...	...	...	Annealed, coiled
Inconel X	29.25	56	10.5	10.1	9.7	8.9	7.7	Cold drawn, coiled Aged, 1350 F, 6 hr
16-25-6	28	...	...	...	...	...	...	Cold drawn, coiled
16-25-6	28	...	...	...	...	...	...	Cold drawn, coiled Aged, 1125 F, 24 hr
Elgiloy	32	60	11.1	10.8	10.2	9.2	8.2	Cold drawn, coiled Aged, 1100 F, 10 to 18 hr
Elgiloy	32	...	...	...	...	...	...	Cold drawn, coiled Aged, 1400 F, 20 hr (For tests at 1400 F)
19-9 DX	29.5	...	...	...	...	...	...	Cold drawn, coiled Aged, 1100 F, 1 hr
A-286	22.0	...	...	...	...	...	...	Solution treated, coiled Aged, 1400 F, 16 to 20 hr
S-816	32.5	...	12.3	11.1	11.3	10.7	9.9	Solution treated, coiled Aged, 1400 F, 16 to 20 hr

or recommended treatment temperatures below the proposed test temperature. In these cases, treatments that were believed to be suitable were developed in the laboratory. The processing treatments given to the springs and their respective spring scales, in pounds per inch of deflection are given in Table II.

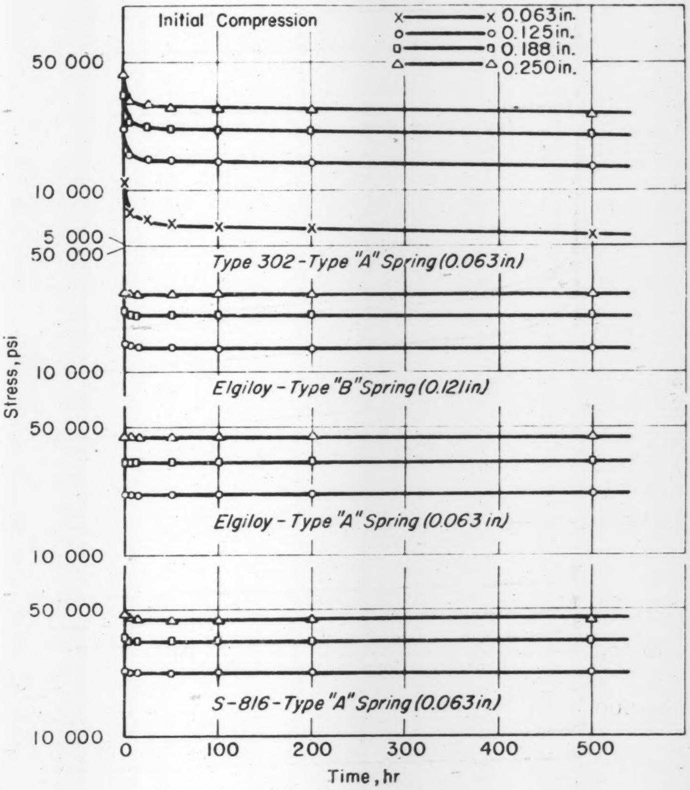


FIG. 2.—Spring Relaxation Tests at 600 F.

For some of the materials, torsional moduli of elasticity were determined at high temperatures by a torsion pendulum method with results given in Table II. Neither the testing equipment nor test specimens are now available for modulus measurements on the other materials investigated. For uniformity, all stress values have been reported on the basis of room-temperature moduli. The correction for modulus variations is only moderate and does not significantly affect the relative ratings of the materials.

RELAXATION TEST PROCEDURE

In the relaxation test, the free length of each spring was measured. The spring was then assembled on a bolt between washers, and a nut was screwed onto the bolt until the desired compression of the spring was reached. The free length of each spring was again determined after exposure to the desired test temperature for total times of 5, 25, 50, 100, 200, and 500 hr.

The effective residual stresses in the springs were determined by the formula:

$$S = \frac{8DCFK}{\pi d^3}$$

where:

S = stress, psi,

D = mean diameter of the spring, in.,

C = spring scale (lb per in. of deflection),

F = elastic deflection of the spring at the end of test period, in.,
d = spring wire diameter, in., and
K = Wahl stress correction factor.

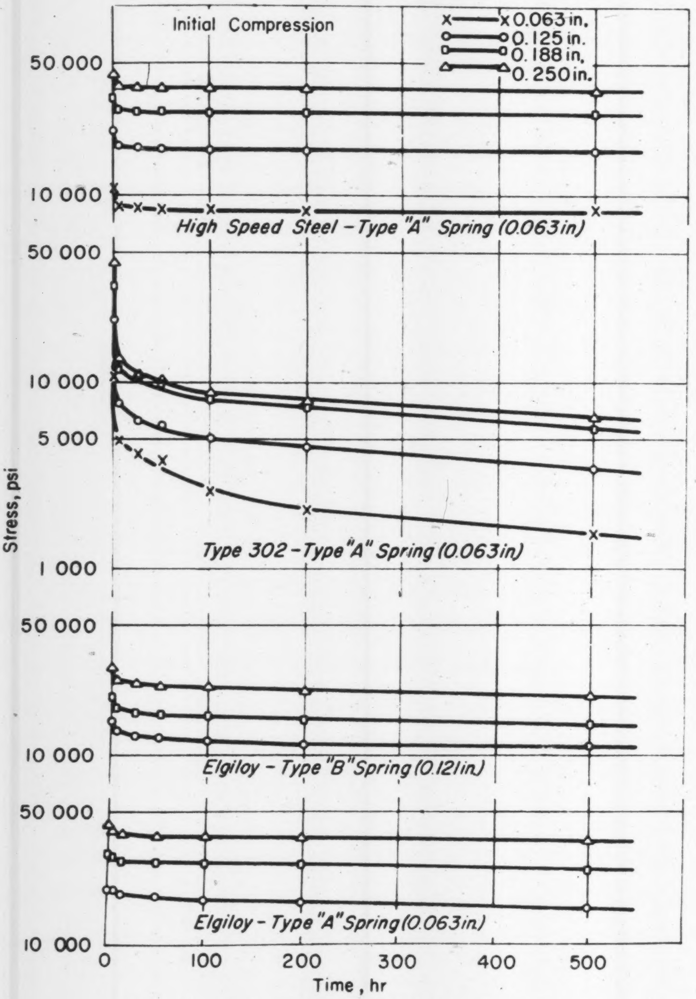


FIG. 3.—Spring Relaxation Tests at 800 F.

RESULTS

All of the spring materials relaxed rapidly in the early stages of the test. As the tests proceeded, the rates of relaxation of most spring materials decreased markedly. The most satisfactory of several methods tried for graphica presentation of the data was a semilogarithmic plot in which stress was plotted on the logarithmic scale and the time of test on a linear scale. The experimental results obtained are presented in a series of such semilogarithmic graphs in Figs. 2 to 12, inclusive.

The materials tested at 600 F were type 302 stainless steel, Elgiloy, and

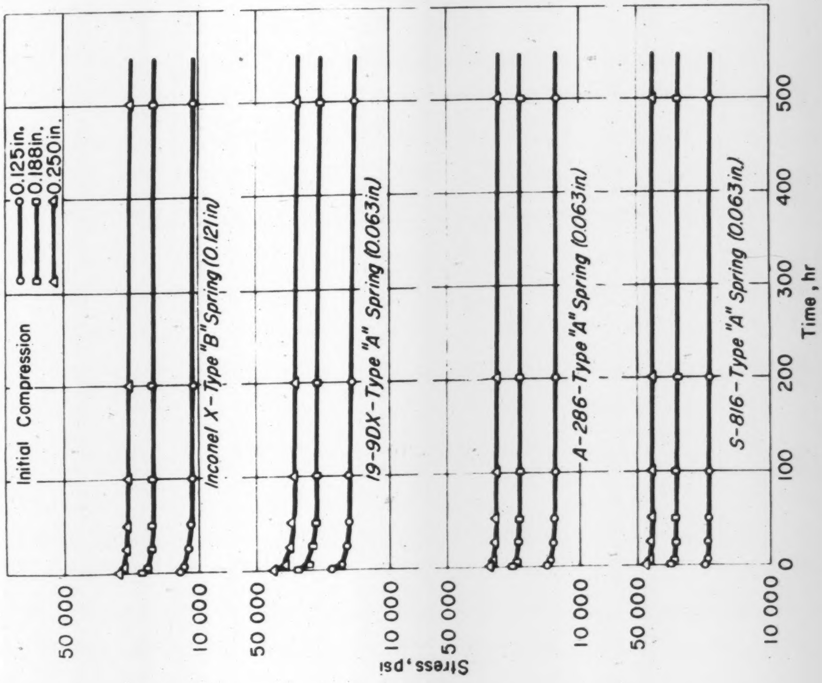


FIG. 4.—Spring Relaxation Tests at 800 F (Continued).

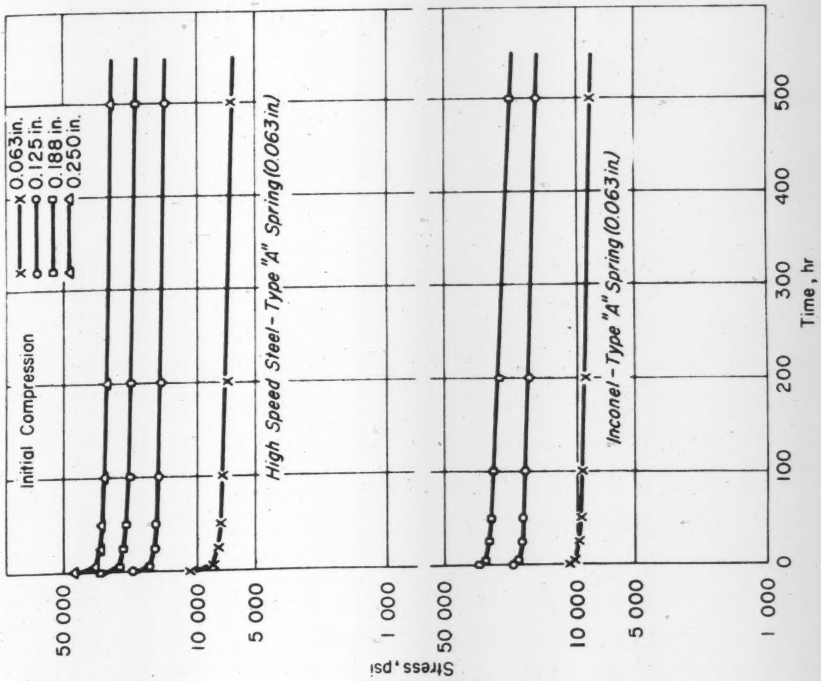


FIG. 5.—Spring Relaxation Tests at 900 F.

S-816. These test results are presented in Fig. 2. The type 302 stainless steel springs relaxed rapidly during the first 25 hr. of test and then relaxed slowly. Elgiloy springs and S-816 springs did not relax appreciably at this temperature.

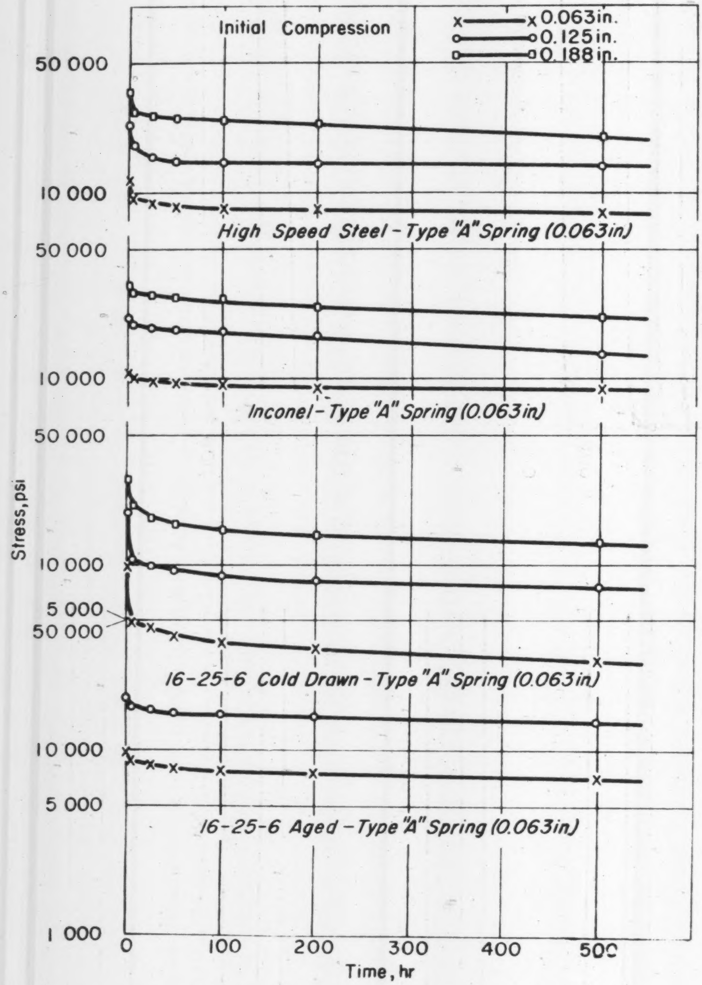


FIG. 6.—Spring Relaxation Tests at 950 F.

The relaxation characteristics of high-speed steel, type 302 stainless steel, Elgiloy, Inconel X, 19-9-DX, A-286, and S-816 were determined at 800 F (Figs. 3 and 4). At this temperature, high-speed steel relaxed slightly during the first 25 hr of the test, after which time relaxation practically stopped. The type 302 stainless steel relaxed very rapidly during the first 100 hr of the test at 800 F, then continued to relax rapidly for the remainder of the test. The Elgiloy springs relaxed slightly for the first 25 hr of test at 800 F, then relaxed at a moderate rate for the remainder of the test. It should be noted that the correlation between

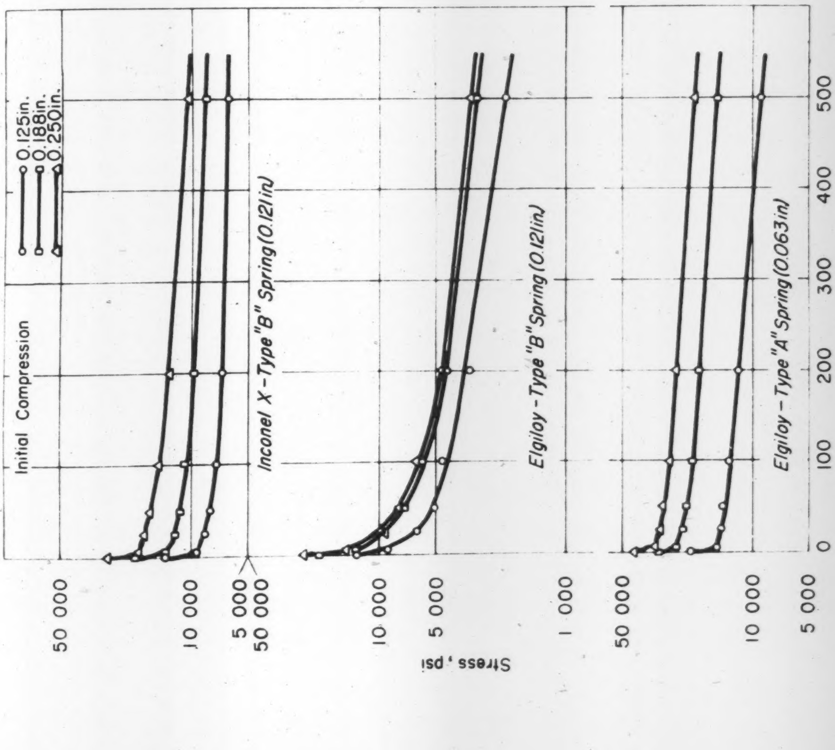


FIG. 7.—Spring Relaxation Tests at 1000 F.

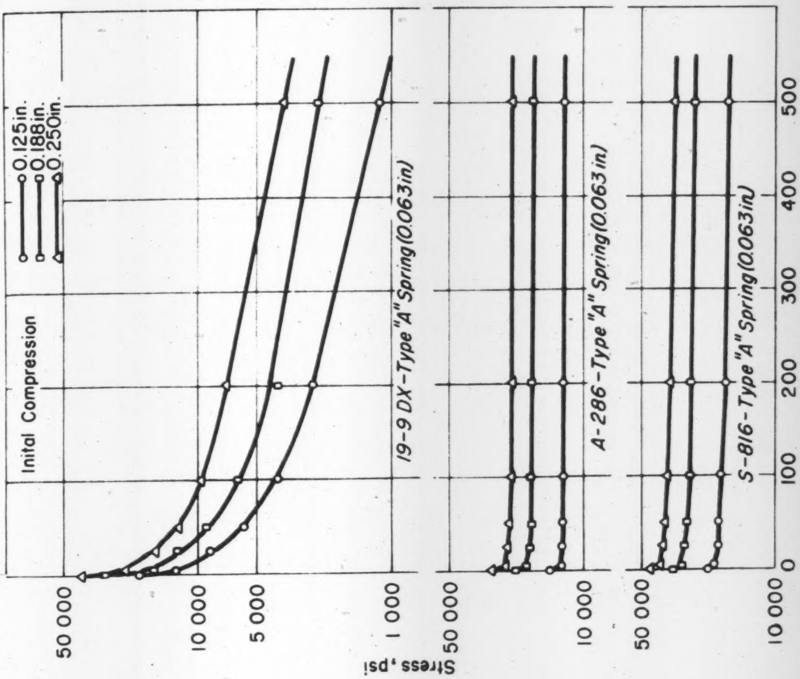


FIG. 8.—Spring Relaxation Tests at 1000 F (Continued).

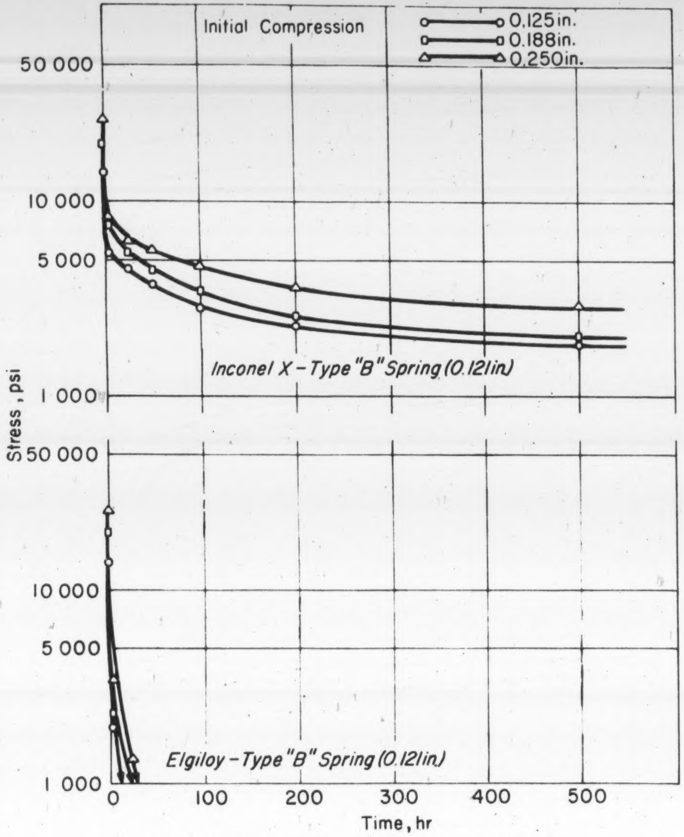


FIG. 9.—Spring Relaxation Tests at 1200 F.

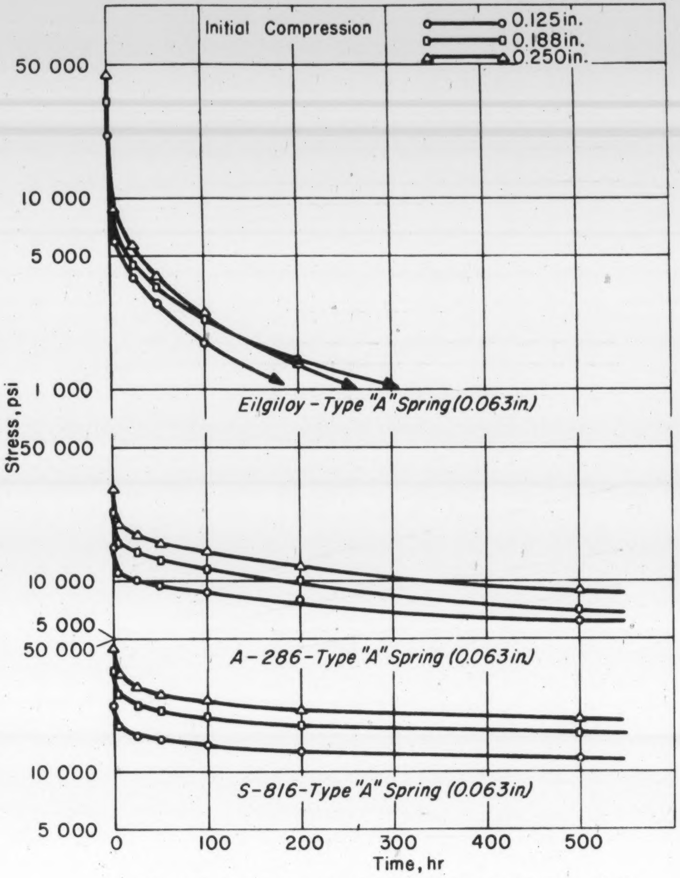


FIG. 10.—Spring Relaxation Tests at 1200 F (Continued).

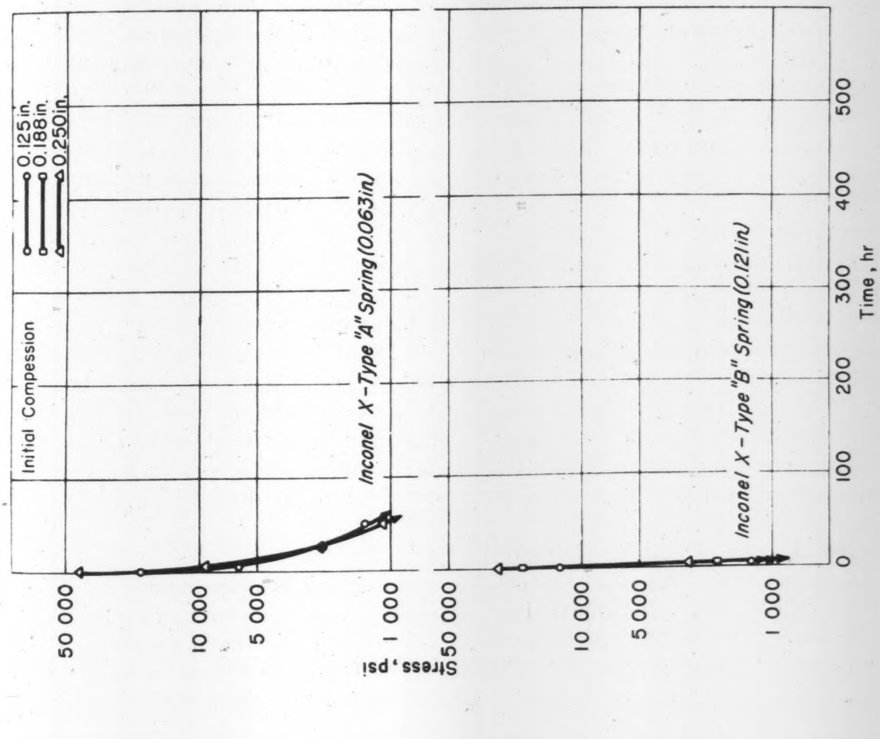


FIG. 11.—Spring Relaxation Tests at 1400 F.

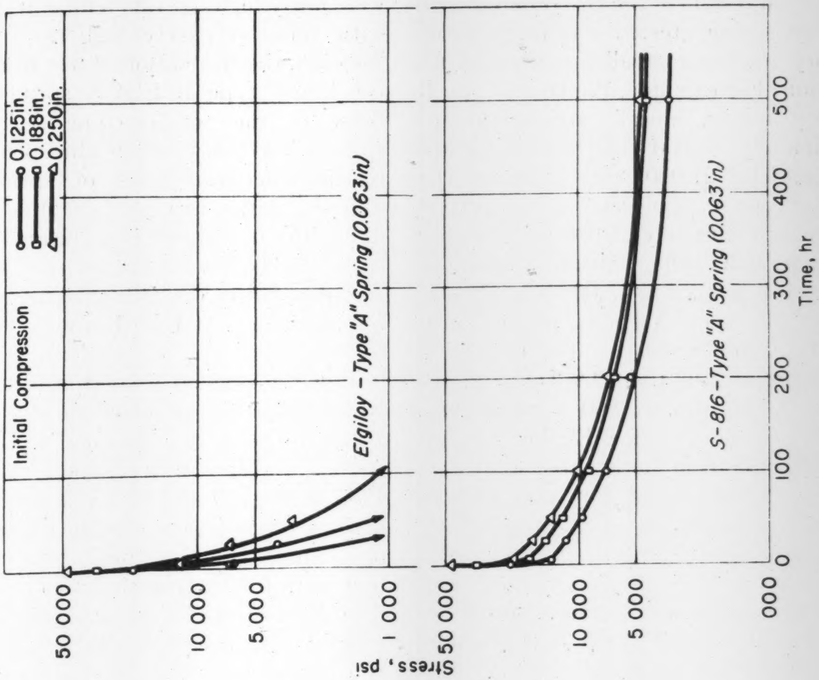


FIG. 12.—Spring Relaxation Tests at 1400 F (Continued).

relaxation results of the two different size Elgiloy springs tested was fairly satisfactory for these small amounts of relaxation. For example, the larger type B Elgiloy spring, initially stressed at approximately 30,000 psi, relaxed to approximately 21,000 psi, whereas the smaller type A spring relaxed from 30,000 psi to approximately 25,000 psi. Another larger type B spring, initially stressed at 20,000 psi, relaxed to 15,000 psi, whereas the smaller type A spring relaxed from 20,000 psi to 15,500 psi. The relaxation characteristics of the 19-9 DX and Inconel X springs at 800 F were comparable to those of the Elgiloy springs. The precipitation hardenable alloys A-286 and S-816 relaxed only very slightly in the initial stages of the test at 800 F, after which relaxation practically stopped.

Only two materials were tested at 900 F, high-speed steel and Inconel (Fig. 5). Both of these materials relaxed a moderate amount at the beginning of the test and continued to relax slowly during the entire test.

The relaxation characteristics of high-speed steel, Inconel, and of Timken 16-25-6 were determined at 950 F (Fig. 6). All of these materials relaxed at small or moderate rates during the entire test. The desirability of stress relief of cold-drawn materials is evident by comparison of the relaxation characteristics of the Timken 16-25-6 springs tested as coiled from cold-drawn wire with those which were given a preliminary treatment after coiling. Both the initial relaxation and the rate of relaxation in the later stages of the test were considerably reduced by the preliminary heat treatment of these springs.

At 1000 F, the relaxation characteristics of Inconel X, Elgiloy, 19-9 DX, A-286, and S-816 were determined (Figs. 7 and 8). Initial and final relaxation rates of the Inconel X, Elgiloy, and 19-9 DX were fairly high. At this temperature and with these relatively high rates of relaxation, the correlation of test results on the larger type B Elgiloy springs with those of the smaller type A Elgiloy springs was very poor. This poor correlation between tests of springs of different design is a good example of the difficulties introduced into the analysis of spring relaxation data because of the complex stress distributions introduced by relaxation. At 1000 F, the A-286 and S-816 springs relaxed a moderate amount during the first 50 hr of the test, after which relaxation occurred slowly.

Relaxation tests of Inconel X, Elgiloy, A-286, and S-816 were performed at 1200 F (Figs. 9 and 10). The Inconel X springs relaxed very rapidly in the first few hours, then continued to relax at a high rate for the remainder of the test. The Elgiloy springs relaxed extremely rapidly and are considered to be practically useless at this temperature. The A-286 springs underwent a moderate amount of initial relaxation, then continued to relax at a moderate rate. The initial relaxation of the S-816 springs was approximately the same as that of the A-286 spring, but in the later stages of the test, the relaxation rate of the S-816 springs was considerably lower than that of any other spring material tested at 1200 F.

Inconel X, Elgiloy, and S-816 materials were tested at 1400 F (Figs. 11 and 12). Of these materials, only S-816 shows any tendency for spring behavior at this temperature. Even for the S-816 springs, the initial relaxation was very great and a stable relaxation rate was not established during the 500-hr test.

SUMMARY

The relaxation characteristics of compression springs under constant deflection loading have been presented in this paper. The results have been presented on the basis of an effective residual stress calculated from the recovery of the spring when the restraint was released. It is believed that this effective residual stress value is useful for design purposes under conditions of only moderate relaxation. Because of the complex stress distribution introduced by appreciable amounts of relaxation, the results presented cannot be applied directly to springs of greatly different size than the ones under test.

Of the nine materials tested, S-816 had the best resistance to relaxation. It is considered that S-816 springs would be useful up to approximately 1200 F. At 1000 F, the relaxation resistance of Inconel X and of A-286 approached that of the S-816 material. At still lower temperatures, severly cold-drawn alloyed austenitic steels also have useful spring properties. If severely cold-drawn austenitic materials are used for high-temperature springs, a preliminary aging or stress relief treatment is desirable in order to reduce the initial relaxation and the final rate of relaxation of these springs in service.

REFERENCES

(1) F. P. Zimmerli, "Effect of Temperature on Coiled Steel Springs Under Various Loading," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 63, May 1941, pp. 363–368.

(2) B. B. Betty, E. C. MacQueen and Carl Rolle, "Relaxation Resistance of Nickel-Alloy Springs," *Transactions*, Am. Soc. Mechanical Engrs., pp. 465–474 (1942).

(3) F. P. Zimmerli and W. P. Wood, "Effect of Temperature on Endurance Limit and Relaxation of Spring Materials," *Transactions*, Soc. Automotive Engrs., Vol. 62, pp. 57–565 (1956).

DISCUSSION

MR. E. A. STICHA.[1] (*presented in written form*).—The authors are to be commended for providing engineers with much useful information in a field where few data are available. At Crane Co. there has been occasion to conduct tests on springs at elevated temperatures also and essentially the same technique as that of Messrs. Matters and Lochen has been employed. Results are presented for one of the materials reported by the authors together with test data on two other materials, one of which appears to be the equal at 1100 F of their best alloy, S 816.

The Crane Co. spring relaxation test results are shown in the accompanying Table III and in Fig. 13, are compared with data taken from the paper. The method of comparison is one frequently employed in assessing high-temperature bolting materials. The 45-deg line represents an ideal material wherein the applied stress is retained completely; proximity to this line is a measure of the worth of a material as a spring. This chart provides a convenient means of correlating tests run at different stress levels under constant temperature from which other useful graphs may be prepared.

Of particular interest is the single Crane test point for an Inconel X spring at 1100 F which is consistent with the Allis-Chalmers results at 1000 and 1200 F. Data for type 316 springs at 1100 F show a marked lack of spring properties for this material. Haynes Stellite No. 25 alloy, on the other hand, retains a considerable portion of the applied stress after 100 hr at 1100 F. These results fall about midway between those for S 816 alloy at 1000 and 1200 F indicating approximate equivalence of relaxation resistance for the two materials. Some test points for the Haynes Stellite No. 25 alloy lie below the line shown in the figure, and this is probably due to the fact that the work was done with experimentally wound springs which were not entirely uniform.

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[1] Crane Co., Chicago, Ill.

TABLE III.—SPRING RELAXATION TEST RESULTS AT 1100 F.

NOTE.—Symbols and method of calculating stress are the same as those employed by Messrs. Matters and Lochen.

Alloy	Mean Spring Diameter, in.	Wire Diameter, in.	Constant, C (D/d)	Constant, K	Initial Stress, psi	100 hr Residual Stress, psi
Inconel X	0.625	0.125	5.00	1.31	83 000	31 500
Type 316	0.625	0.125	5.00	1.31	78 000	13 000
	1.250	0.250	5.00	1.31	64 000	8 000[a]
Haynes Stellite No. 25	1.281	0.250	5.12	1.30	78 500	50 000
	1.281	0.250	5.12	1.30	69 000	41 000
	1.563	0.250	6.25	1.23	22 000	17 000
	0.666	0.125	5.33	1.29	74 000	28 500
	0.875	0.125	7.00	1.21	33 000	11 500

[a] Extrapolated values.

In connection with a recently contemplated design for which materials were restricted, a relaxation test on a molybdenum wire spring at 1050 F revealed disappointing load retention in spite of the outstanding creep resistance of molybdenum at much higher temperatures. The reason for this deficiency is not clear, but it may be due to a low proportional limit in torsional shear; this unexpected behavior is being studied further.

MR. G. N. AGGEN[2] (*by letter*).—The authors are to be congratulated upon an interesting and informative paper. In two cases, the authors obtained results which do not appear to correlate with other known mechanical properties of the alloys tested.

For one, the authors assigned a spring scale of only 22.0 lb per in. to A-286 alloy while those of the remaining alloys varied from 28 to 34. Since the springs were presumably deformed elastically, the resulting spring scales should be directly proportional to the torsional moduli. The moduli reported for two of the alloys are as follows:

TORSIONAL MODULUS, PSI, AT INDICATED TEMPERATURES.

Alloy	80 F	1000 F	1200 F	1400 F
A-286[a]......	10.4×10^6	8.4×10^6	8.0×10^6	7.5×10^6
Inconel X[b]...	11.0×10^6	9.0×10^6	8.1×10^6	...

[a] Allegheny Ludlum Technical Data Sheet.

[b] Inconel X Data Sheet, International Nickel Co., Inc., 1947.

These values indicate that the spring scale of A-286 should be only slightly lower than that of Inconel X. On the basis of similarities in composition and structure also, the spring scale of A-286 would be expected to be comparable to those of 19-9DX and 16-25-6, or about 28.0 lb per in. of deflection.

Secondly, the relaxation rates obtained on the Inconel X springs at 1200 and 1400 F are surprisingly rapid when compared to A-286 and S-816 at 1200 F and S-816 at 1400 F. The creep strengths reported in ASTM Special Technical Publication No. 160[3] for these alloys

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[2] Senior Research Metallurgist, Research Laboratory, Allegheny Ludlum Steel Corp., Watervliet, N. Y.

[3] Report on Elevated Temperatures Prop-

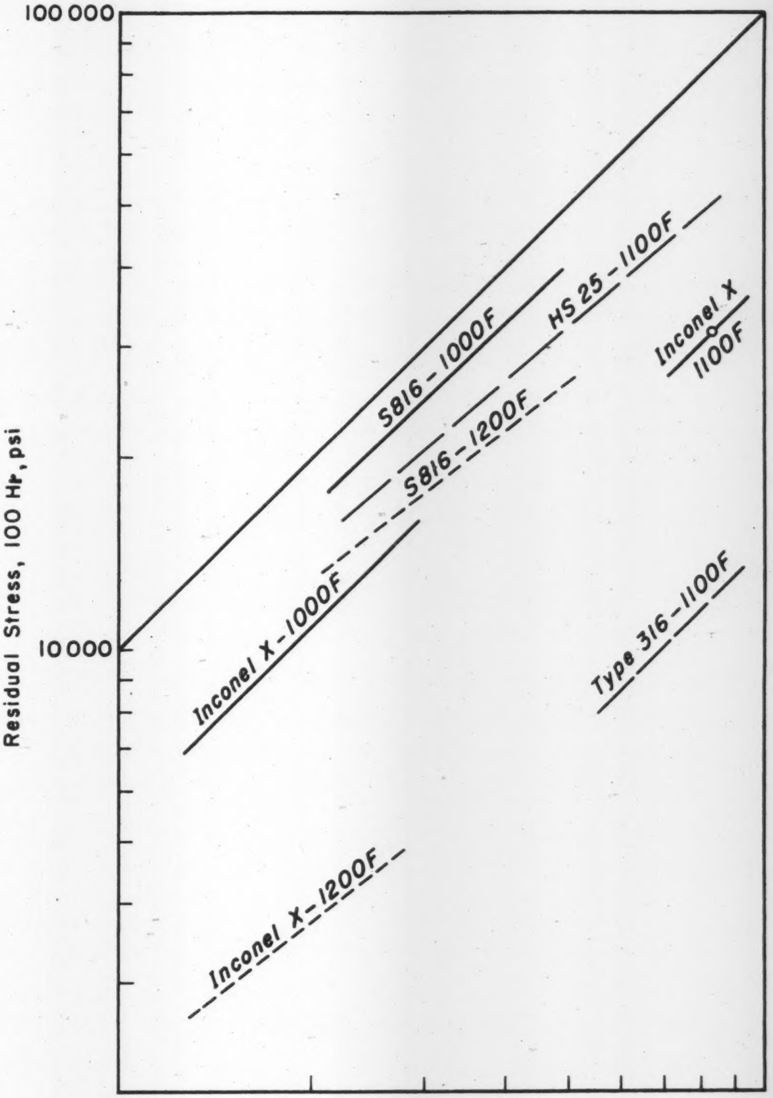

FIG. 13—Comparison of Spring Relaxation Properties of Several Alloys.

follow:

CREEP STRENGTH, PSI, FOR CREEP OF 0.0001 PER CENT PER HR.

Alloy	1200 F	1400 F
A-286	28 000	...
S-816	28 000	16 500
Inconel X	63 000	30 000

A direct comparison at 1200 F is not possible since the Inconel X was tested as a type B spring and the other alloys as type A springs. The authors have already pointed out the poor correlation

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erties on Selected Superstrength Alloys (1954). (Issued as separate publication *ASTM STP No. 160.*)

obtained between Elgiloy springs of the two different types at 1000 F. A contributing cause for the comparatively poor behavior of Inconel X is very probably the difference in spring design.

At 1400 F, where both Inconel X and S-816 were tested as type A springs, the S-816 alloy was far superior. On the basis of creep strengths, the reverse would be expected, particularly since good correlations are generally obtained between creep and creep relaxation strengths.

The authors imply, during their discussion of processing and heat treatment, that the S-816 and Inconel X were both cold drawn and aged directly. If this is the case, Inconel X, which is predominantly a precipitation-strengthened alloy, may have been overaged before testing or during test. For example, the aging temperature to produce maximum hardness in A-286 is about 1300 F for solution heat-treated material and drops to about 1100 F for material that has been cold worked 80 per cent. Inconel X is expected to show similar behavior. S-816, on the other hand, is a solution-strengthened alloy. Cold work followed by aging would not produce softening unless the recrystallization or recovery temperatures were exceeded. For S-816, these temperatures are believed to be above 1400 F even with large amounts of previous cold work.

Similarly, it is believed that improved properties would have been obtained from the A-286 alloy if a lower aging temperature had been selected. For maximum hardness and creep strength, the aging temperature should be about 1325 F for solution heat-treated material and lower for cold-worked material, depending upon the amount of cold deformation.

MR. R. G. MATTERS (*author's closure*).—We are pleased to know that Mr. Sticha's relaxation results are consistent with ours. The consistency of the results is largely dependent upon the fact that the materials and test temperatures were such that large amounts of relaxation did not occur. As indicated in the paper, the residual stress reported is based upon an elastic recovery of the spring. If appreciable relaxation occurs during the test, the reported residual stress is not simply related to the actual stresses, and correlation between tests made on springs of different designs could not be expected.

With reference to Mr. Aggen's discussion, we cannot account for the discrepancy in spring scales of springs made from different materials. The spring scales reported were based upon measurements made with a commercial spring tester.

It is now recognized that some of the materials tested probably were not processed in an optimum manner. The Inconel X springs were simply coiled from cold-drawn wire and aged. This processing was recommended by the International Nickel Co. in 1948 when these tests were started. Probably the Inconel X springs would have had better relaxation resistance if they had been coiled from solution-treated wire. S 816 and A 286 springs were coiled from solution-treated wire rather than cold-drawn wire. The aging temperature, 1400 F, for the A 286 springs was the highest temperature which, based on hardness tests, did not cause over aging of the particular lot of wire used.

Based upon the test results available, it is believed that springs for the highest temperature application should be coiled from solution-treated super alloy wire, then stress relieved or aged at the highest practical temperature in order to relieve the coiling stresses. It is apparent that cold-drawn materials are limited to somewhat lower temperature service. Possibly solution treatment of the springs after the coiling operation followed by aging would develop the maximum resistance to high temperature relaxation.

THE STRENGTH OF CERTAIN STAINLESS AND CARBON STEELS AT LOW TEMPERATURES*†

BY S. C. COLLINS,[1] F. D. EZEKIEL,[2] O. W. SEPP[3] AND J. W. RIZIKA[4]

SYNOPSIS

Preliminary results of torsion and tension tests of various steels at very-low-temperatures are reported. The tests are conducted in a stream of cold gaseous helium whose temperature can be adjusted to any value between 10 and 300 K. Unusual discontinuities in the stress-strain relationship of stainless steel at the lower temperatures are observed. The effect of work hardening at low temperature upon the room-temperature strength is also noted.

It has long been known that as the temperature of a metal decreases its tensile strength increases and that some metals such as brass, copper, and many types of stainless steels remain plastic down to temperatures near the absolute temperature while others such as ordinary carbon steels become brittle at temperatures far above the absolute zero. A large body of data on the mechanical properties of metals at low temperatures has accumulated as a result of the efforts of numerous investigators. Stainless steels in particular have been studied by McAdam, Geil, and Cromwell (**1**)[5] who showed how the load during a tension test of 18-8 stainless steel varies with strain at various low temperatures, and by Krivobok and Talbot (**2**) and Ziegler and Brace (**3**) who demonstrated that austenitic steels mechanically worked at liquid nitrogen temperature show an increase in tensile strength, yield strength, and hardness as compared with steels worked at room temperature. Nearly all of their measurements were made at a few specific temperatures, namely, the sublimation temperature of carbon dioxide (194.6 K), the boiling point of oxygen (90.1 K) or the boiling point of nitrogen (77.3 K). The reason for choosing these particular temperatures is, of course, their ready availability. The results reported below constitute an extension of the work of these investigators and are distributed over the temperature range 300 K to 10 K without regard to the usual fixed points. The test specimens are cooled by a stream of cold gas rather than by im-

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[4] Supervisor of Turboconditioner Cycle and Systems Design Unit, Aircraft Accessory Turbine Dept., General Electric Company, Lynn, Mass.

[5] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 699.

mersion in a liquid. Earlier work of this kind, limited to 1020 steel, has been reported by Eldin and Collins (**4**).

Apparatus and Methods

Two types of tests were made in this investigation, torsion and tension, the quantities measured being torque or load and its accompanying strain. The cold environment was provided by a helium cryostat. This consists of a vacuum jacketed cylindrical vessel with an inside diameter of 30 in. and a depth of 8 ft, open at the top. A part of the space is occupied by a heat exchanger and five expansion engines. Helium is compressed by an external compressor to 10 atmospheres, cooled to room temperature, and supplied to the expansion engines by way of one channel of the counter-flow heat exchanger. Upon expansion to one atmosphere with the production of external work, the helium leaves the expansion engines very much colder and returns to the compressor by way of a second channel in the heat exchanger. When a steady state is reached the engines are operating at various temperatures between ambient and the lowest level. The gas discharged from the coldest engine is forced to flow over and around the specimen under test before it is admitted to the heat exchanger. When the desired temperature is reached, the operating pressure is reduced sufficiently so that the refrigerative effect just compensates for the heat

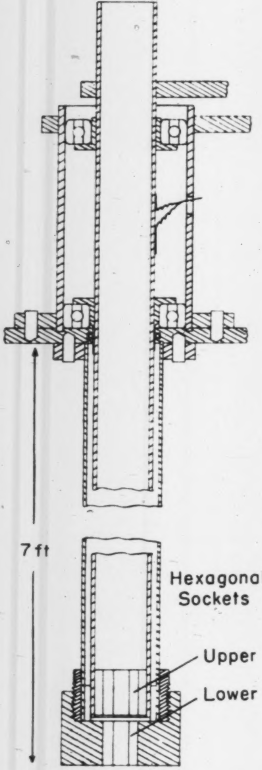

Fig. 1.—Torsion Test Apparatus.

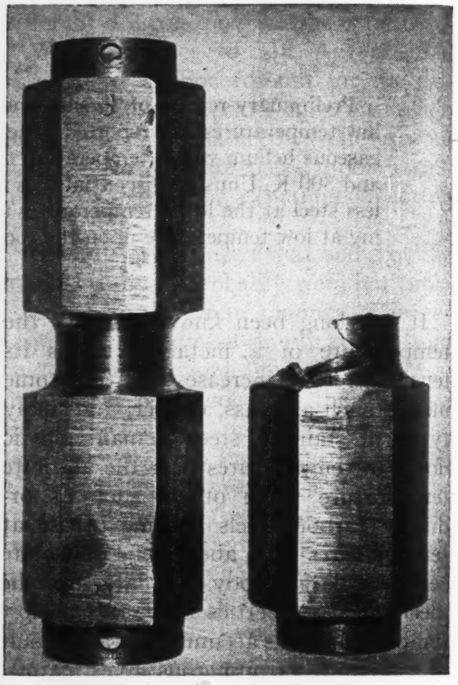
(*a*) (*b*)

Fig. 2.—Torsion Test Specimen.
(*a*) In original condition.
(*b*) After rupture at 30 deg Kelvin.

leaks. It is possible to keep the temperature fairly constant by manual control of the operating pressure.

Both the torsion and tension testing apparatus are mounted on steel plates which act as gas-tight covers for the cryostat, the test apparatus extending deep into the cold well. The torsion equipment is made of two concentric stainless steel tubes arranged as indicated in Fig. 1. The lower end of each tube is fitted with an hexagonal socket for holding the two ends of the test specimen. Ball bearings, at the warm end, and a sleeve bearing of micarta, at the cold end, in the annular space between the tubes maintain true alignment and reduce friction when the inner tube is rotated through the angle necessary to break the specimen. A specimen for use in this machine is shown in Fig. 2(*a*). It is made from a 1-in. hexagonal bar with a $\frac{9}{16}$-in. hole along the axis. The test section is reduced to a short tube $\frac{1}{2}$ in. long, 0.73 in. outside diameter and 0.63 in. inside diameter.

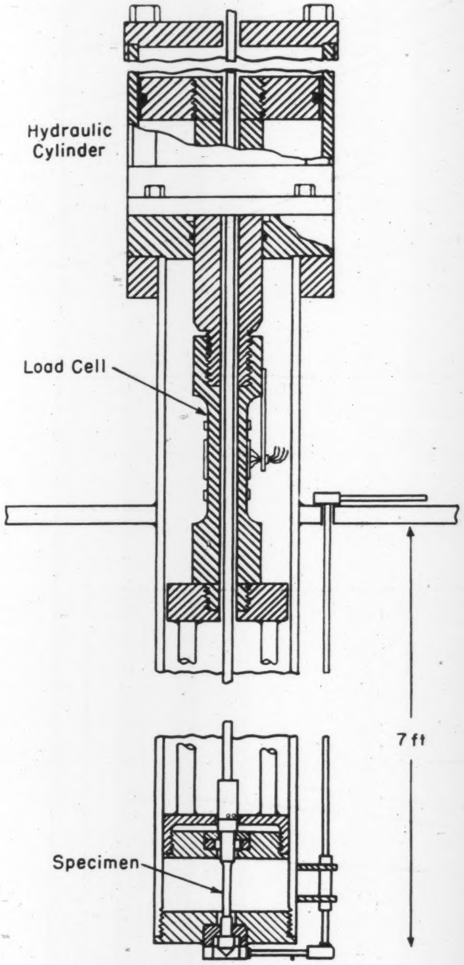

FIG. 3.—Tensile Testing Machine.

The tension testing machine is shown in Fig. 3. The compression member is an 8-ft length of stainless steel pipe with a 6-in. outside diameter and $\frac{1}{4}$ in. wall. The specimen is held in the lower end and therefore at the bottom of the cold well. The jaws which hold the lower end of the specimen are controlled by rods which extend through the cover to the exterior. Force is applied to the specimen by a hydraulic piston mounted at the upper end of the compression member through stainless steel pull rods. Stainless steel is chosen for all structural parts of the apparatus because of its low thermal conductivity. Moreover, it is very strong. Specimens can be inserted or withdrawn through the hollow piston rod. The test section of the specimen is 2 in. long and 0.305 in. in diameter as shown in Fig. 11(*a*).

Temperatures are measured by copper-constantan thermocouples. At the lowest temperatures the accuracy is low, an error of a few degrees being possible. For both torsion and tension tests the thermocouple junction is soldered to a brass receptacle in which one end of the specimen is gripped by set screws. Since the entire specimen holder is bathed in a copious stream of cold helium, there is reason for believing the temperature to be uniform over the specimen and thermocouple junction.

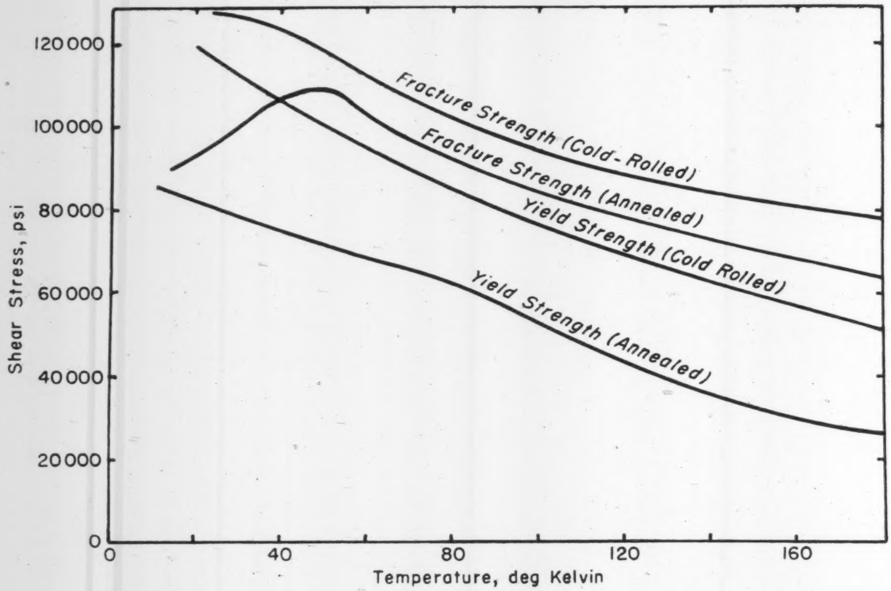

FIG. 4.—Fracture and Yield Stress of 1018 Steel in Annealed and Cold-Rolled Condition as a Function of Temperature.

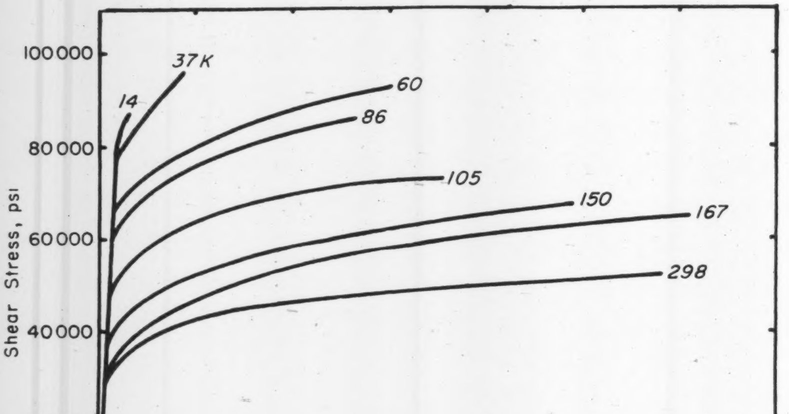

FIG. 5.—Stress-Strain Curves for 1018 Steel in Torsion at Various Temperatures.

Torque is measured by means of strain gages appropriately mounted on a load cell interposed in the inner torque member. The load cell was previously calibrated against known torques. The shear stress is estimated from measurements of torque and inside and outside diameters of the specimen. Shear strain is estimated from measurements of the angle through which one end of the specimen is turned with respect to the other end, the diameter and the length of the test section.

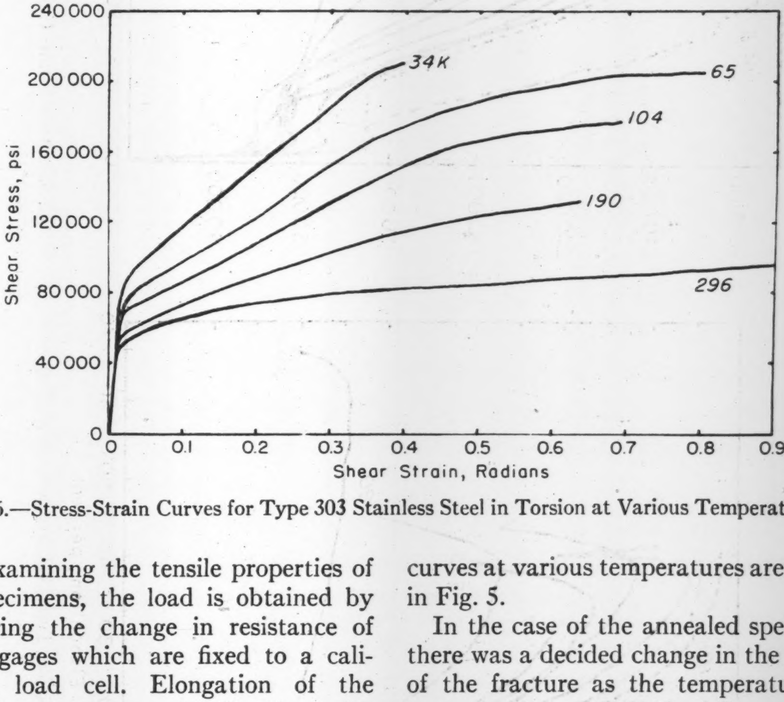

FIG. 6.—Stress-Strain Curves for Type 303 Stainless Steel in Torsion at Various Temperatures.

In examining the tensile properties of the specimens, the load is obtained by measuring the change in resistance of strain gages which are fixed to a calibrated load cell. Elongation of the specimen is observed directly with telescope and scale placed at the top of the specimen holder. In the present form of the apparatus it is not possible to follow the changes in specimen diameter during the test.

All of the specimens were cut from commercial stock and tested in the "as-received" condition with the exception of the 1018 annealed batch. These were given an additional annealing treatment. The chemical composition is not accurately known.

TEST RESULTS

For the torsion tests three types of steel were used, 1018 annealed, 1018 cold-rolled, and 303 (stainless) annealed.

The yield and fracture stresses of 1018 steel both in the annealed and cold-rolled condition are given as a function of temperature in Fig. 4. The stress-strain curves at various temperatures are shown in Fig. 5.

In the case of the annealed specimens there was a decided change in the nature of the fracture as the temperature decreased. Above 130 K the failure was the result of pure shear. The polished surface of the test section was dulled by yielding and the parting line lay in a plane perpendicular to the axis. Below 80 K brittle fracture always occurred, generally along a line inclined 45 deg from the axis. At the lowest temperatures cracks, originating in the relatively thin

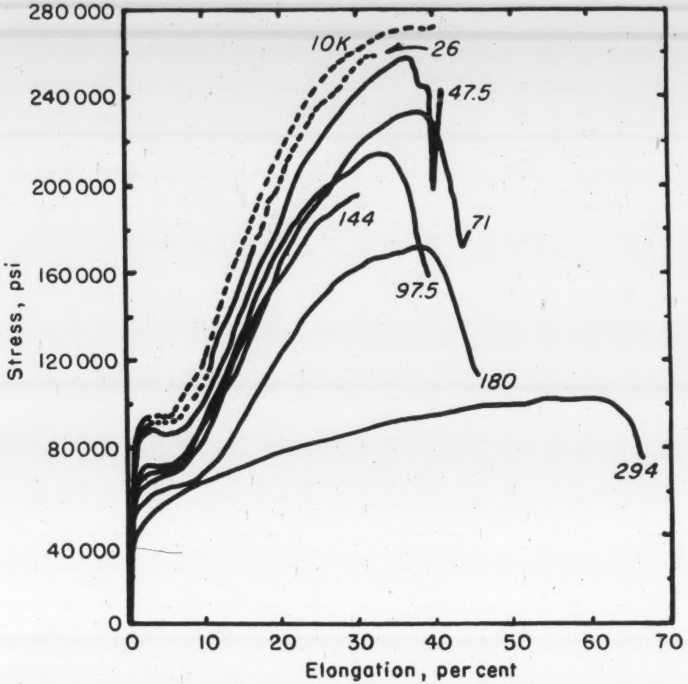

FIG. 7.—Stress-Strain Curves for Type 302 in Tension at Various Temperatures.

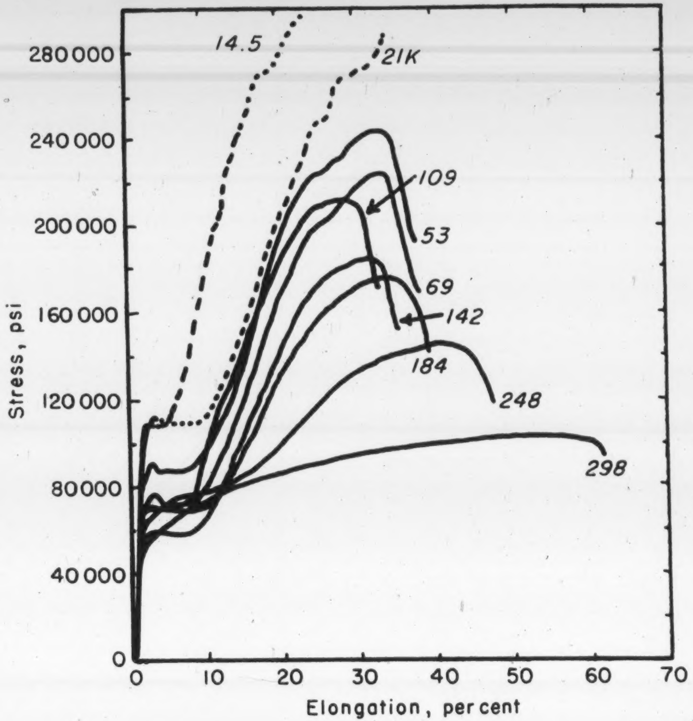

FIG. 8.—Stress-Strain Curves for Type 303 in Tension at Various Temperatures.

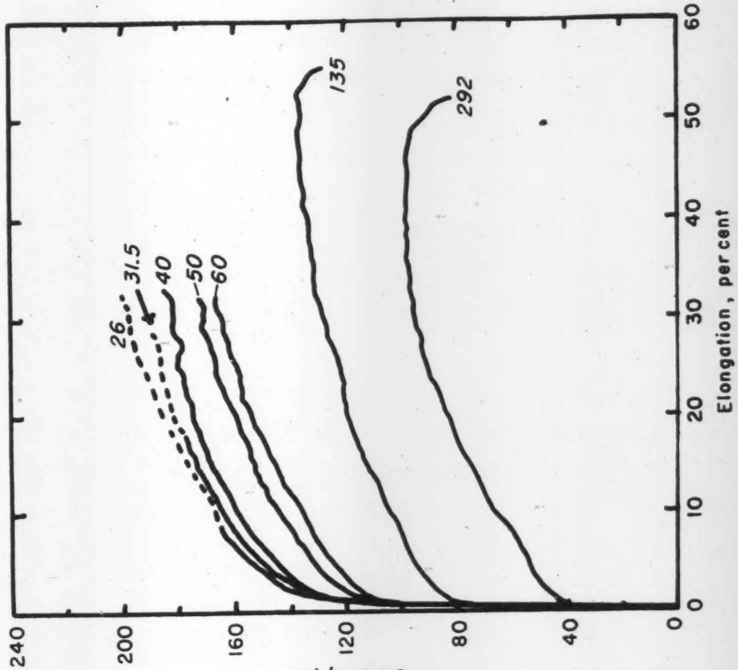

FIG. 9.—Stress-Strain Curves for Type 310 in Tension at Various Temperatures.

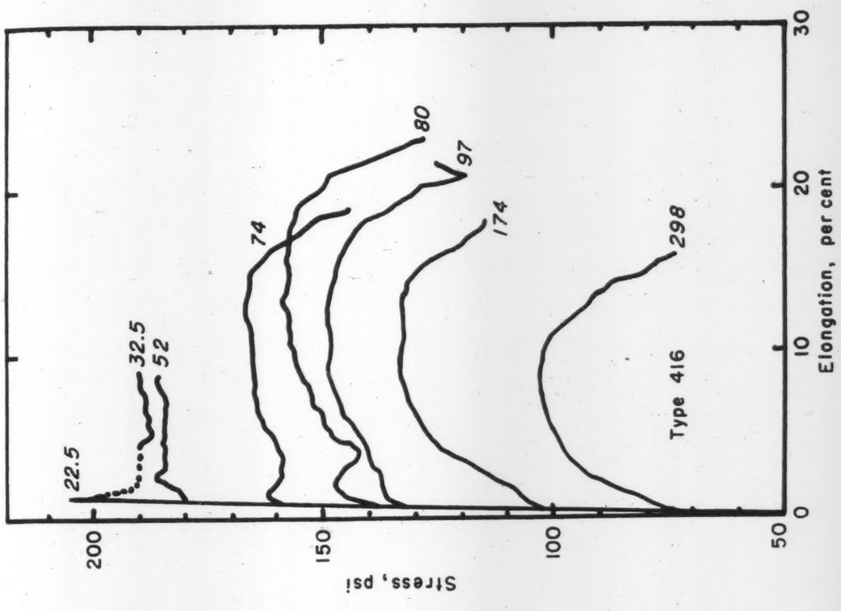

FIG. 10.—Stress-Strain Curves for Type 416 in Tension at Various Temperatures.

wall of the test section, penetrated the thicker shoulders of the hexagon as illustrated by Fig. 2(*b*). Between 80 and 130 K failure was partially ductile, partially brittle.

Cold-rolled specimens, as expected, exhibited definitely higher yield strengths throughout the temperature range, but showed less brittleness. Even at 24 K there was evidence of shear strain. The strain was confined to a narrow band, visible on the inner as well as the outer surface, in which failure ultimately occurred.

The results with type 303 stainless steel are of special interest because of the appearance of discontinuities in the stress-strain relationship at temperatures below 35 K and because of the substantial increase of strength with strain. Some typical stress-strain curves are given in Fig. 6.

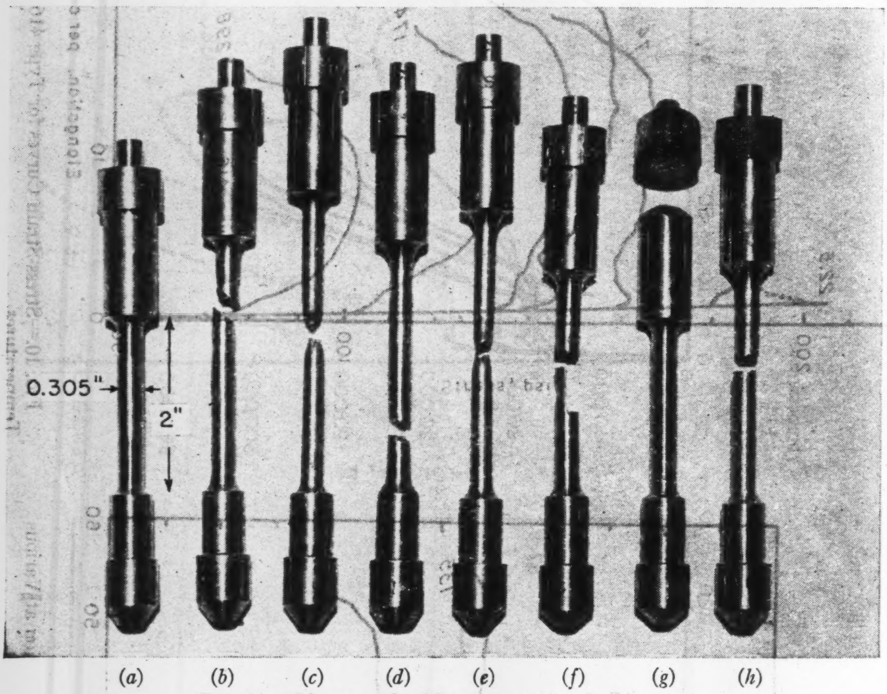

FIG. 11.—Photograph of Representative Specimens.

(*a*) Specimen in initial condition.
(*b*) Type 302, 10 K. Note multiple necks.
(*c*) Type 302, room-temperature.
(*d*) Type 303, 24 K.
(*e*) Type 303, room-temperature.
(*f*) Type 416, 96 K. Note that neck had formed before brittle failure occurred.
(*g*) Type 416, 14 K.
(*h*) Type 310, 17 K. Note beaded surface and substantial elongation without necking.

The discontinuities in the stress-strain relationship referred to above had the appearance, when encountered, of a failure of the specimen in that the load dropped suddenly to a fraction of its original value. Except for the substantial elastic strain of the apparatus, the load would have dropped to zero. Upon further twisting of the specimen, however, the load climbed quickly to the original value and the earlier stress-strain relationship was reestablished. Photomicrographs of sections of specimens behaving in this manner did not show anything which might help to explain this effect.

One specimen whose yield strength at 34 K was 75,000 psi was twisted to a shear strain of 0.14 at that temperature. It was then returned to room temperature. The new yield strength at room temperature was 95,000 psi a number which is to be compared with 45,000 psi, the normal yield strength at room-temperature.

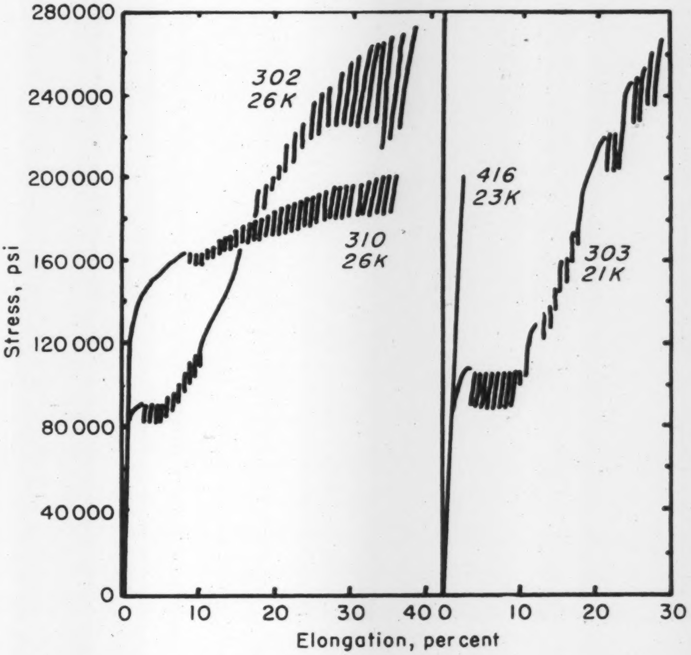

FIG. 12.—Discontinuities in Stress-Strain Curves at Very-Low-Temperatures.

While conducting the tension tests, it was *necessary to avoid brittle metals* with the apparatus in its present form. The grips apply unknown and variable bending moments to the specimens with the result that brittle fracture strength cannot be measured with any accuracy. It is expected that this defect will be corrected soon and that carbon steels may be studied further.

The metals tested in tension to date are three austenitic stainless steels, types 302, 303, 310 and one ferritic, type 416. All of these were commercial grades in the annealed condition as received from the dealer. It is suspected, however, that all had been work-hardened to some extent. Types 302 and 303 are of the 18-8 grade while type 310 contains about 25 per cent chromium and 20 per cent nickel. Type 416 contains about 13 per cent chromium and no nickel.

In Figs. 7, 8, 9, and 10, the nominal stress, obtained by dividing the tensile load by the original cross-sectional area of the specimen, is plotted against per cent elongation for each of the four metals at various temperatures. Types 302 and 303 reveal similar results, as would be expected from their composition. The increase of yield strength with decreasing temperature is more marked for 303 than for 302. Both exhibit substantial elongation in the beginning while the load remains almost constant. Further yielding, however, is attended at reduced temperatures by phenomenal gains in strength. At temperatures above 40 K a single neck eventually forms and the load drops precipitately although the true stress is still rising. At lower temperatures, especially below 30 K multiple necks may, and generally do, occur. (See Figs. 11(*b*) and 11(*d*).) The gain in strength from the work hardening and the hardening caused by phase changes eventually more than compensates for the reduction of area of a neck, thereby slowing down or stopping its development and permitting a new neck to start. When the specimen is finally broken, rupture does not necessarily take place in one of the necks.

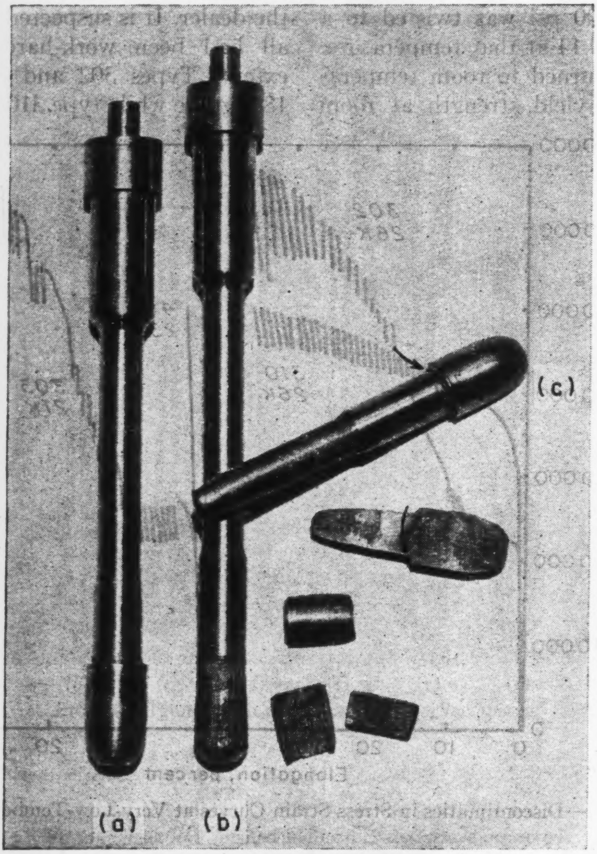

FIG. 13.—Longitudinal Failure, Type 303.

The dotted portions of the curves in Figs. 7, 8, 9, and 10 represent multiple discontinuities of the type noted above as having occurred with stainless steel in torsion. These are shown in greater detail in Fig. 12. The almost vertical-parallel lines are plotted from two or more observations each. They are portions of a stress-strain curve of an elastic material. The upper ends of the lines indicate the maximum stress attained before a catastrophe occurs. The specimen suddenly elongates by a definite and fairly constant amount of 0.005 in. in the beginning and by a larger and more variable quantity toward the end of the test. The lower ends of the lines are not significant. They are determined by the rate at which the load is applied and the "softness" of the apparatus. With a rigid apparatus and a very low strain rate it is assumed that the specimen would be completely unloaded when the slip takes place. The effect was noticed in all four types of stainless steel at very low temperatures, below 45 deg for 303, below 38 deg for 302, below 35 deg for 310, and between 33 and 30 deg for 416. Brittle failure before yielding occurred in type 416 at temperatures below 30 K. The observed increments of length of approximately 0.005 in. are the same for 1-in., 2-in., and 3-in. test sections. This is taken to mean that the stepwise elongation is a localized phenomenon. For a long test section more steps occur for a given percentage elongation. Very high strain rates are attended by rapid oscillation of the stress between high and low values. At lower rates of strain the stress, although a quite irregular function of strain, remains near the peak values obtained at high strain rates.

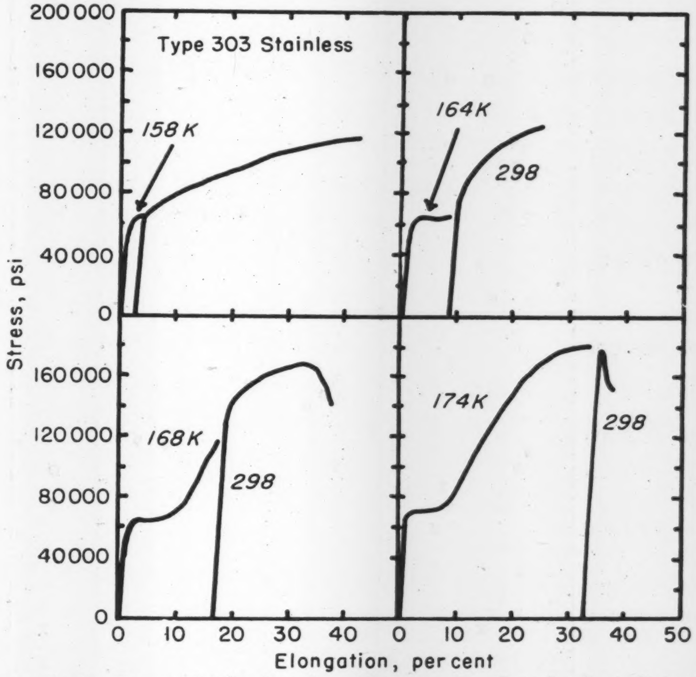

FIG. 14.—Stress-Strain Curves Initiated at Low-Temperature, Completed at Room-Temperature.

In Fig. 11 are shown standard specimens, one in its original condition and others after failure. In Fig. 11(*f*) is seen a specimen of 416 which snapped into three pieces even after necking considerably. Brittle failure in the heavier section is seen in Fig. 11(*g*) (also 416). Type 310 is unusual at low-temperatures for two reasons: although this material is quite ductile no neck is formed and the surface Fig. 11(*h*) appears beaded after appreciable elongation.

In Fig. 13 is shown the result of attempting to break a specimen (303) whose test section was too large. The original form of the specimen is seen in Fig. 13(*a*). In Fig. 13(*b*) is seen the characteristic type of failure at 110 deg K. The shoulder has been sheared from the lower end of the specimen. In Fig. 13(*c*) this type of failure can be seen in an early stage. A crack has started in the fillet and is being propagated in a direction parallel to the axis.

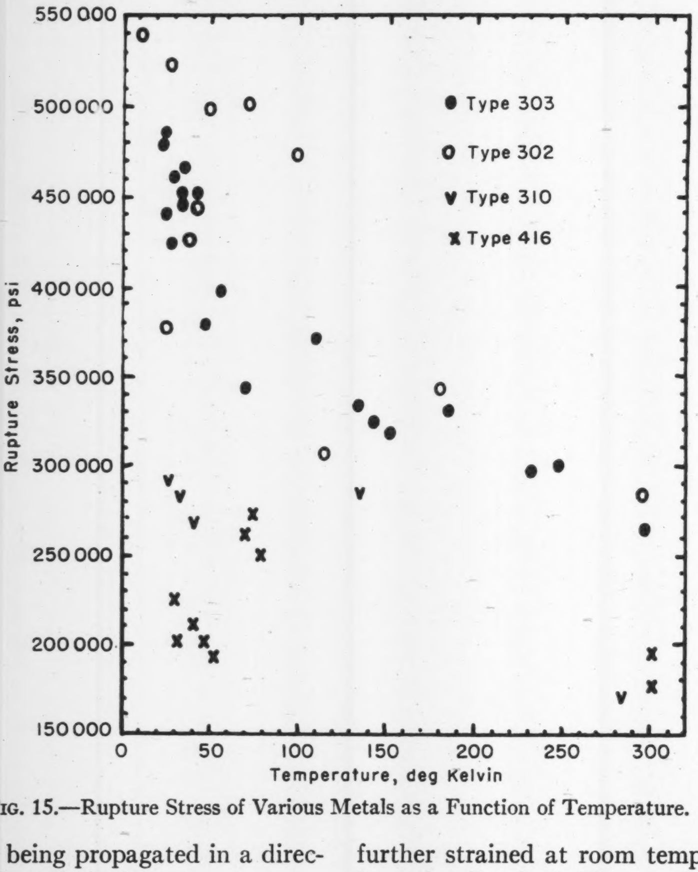

FIG. 15.—Rupture Stress of Various Metals as a Function of Temperature.

The effect of various amounts of low-temperature straining upon the room-temperature properties of stainless steel 303 is presented in Fig. 14. It will be noted that yielding begins at room temperature after a low-temperature treatment, at a stress equal to or greater than the maximum attained during the low-temperature treatment. Reference was made above to the unexpected elongation which is noticed at almost constant stress just at the yield point and to the rapid gain in strength with further strain beyond this stage. The upper right-hand curve in Fig. 14 indicates that a specimen brought to the critical point of maximum strain with no gain in strength at 164 K and further strained at room temperature is least ductile of all. This result has been checked by two additional trials.

Rupture stress (based on the reduced section at rupture) as a function of temperature is given for the four types of stainless steel in Fig. 15. Scattering of results is fairly severe, but impressive magnitudes are generally attained, especially with types 302 and 303.

CONCLUSIONS

Certain steels exhibit at very low temperatures marked discontinuities in the stress-strain relationship especially at high strain rates. The sudden increases in length are fairly uniform in magnitude (0.004 in. 0.005 in.) and independent of the length of the specimen. The specimen may or may not gain in strength after a number of such sudden increases in length.

The improvement in room temperature properties by work hardening at low temperatures depends, in the case of type 303 at least, upon the extent of the work hardening and may actually be negative for intermediate amounts.

For further work it is proposed to make the required changes in the apparatus for testing brittle specimens and to choose material whose composition and history are known. More attention will be given to change in microstructure, density, and resistivity.

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(2) V. N. Krivobok and A. M. Talbot, "Effect of Temperature on the Mechanical Properties, Characteristics and Processing of Austenitic Stainless Steels," *Proceedings*, Am. Soc. Testing Mats, Vol. 50, p. 895 (1950).

(3) N. A. Zeigler and P. H. Brace, "Hardening of Austenitic Stainless Steels by Mechanical Working at Subzero Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 50, p. 861 (1950).

(4) A. S. Eldin and S. C. Collins, "Fracture and Yield Stress of 1020 Steel at Low Temperatures," *Journal of Applied Physics*, Vol. 22, No. 10, pp. 1296-1297 (1951).

DISCUSSION

MR. E. T. WESSEL.[1]—The authors are to be commended for their very interesting paper. I would like to contribute some additional discussion concerning the marked discontinuities in the stress-strain relationship which were observed and described by the authors for the case of the stainless steels tested at low temperatures.

During recent low-temperature mechanical property investigations[2] conducted at the Westinghouse Research Laboratories, marked discontinuities were observed in the autographic stress-strain curves of several metals of various lattice structures (the accompanying Figs. 16 to 20). All tests were conducted in tension at a constant strain rate of 100 per cent per hour (2.8×10^{-4} per sec.) using unnotched specimens and apparatus which is described in the literature.[3] These discontinuities appeared as pronounced repeated yieldings with an elastic loading portion between each consecutive yielding, as was also true for the stainless steels tested by Collins and colleagues. The number of repeated yieldings (serrations), for any one test to fracture varied between metals as well as in any one metal tested at different temperatures (see Figs. 16 to 20). The serrations were most numerous in the nickel and zirconium where the order of 100 yields occurred in each test at 4.2 Kelvin. Contrary to the observations of the present paper, the amount of plastic extension associated with each yielding was observed to increase gradually as straining increased. As the test proceeded, the magnitude of the drop

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[1] Research Engineer, Westinghouse Research Lab., Pittsburgh, Pa.

[2] E. T. Wessel, "Some Exploratory Observations of the Tensile Properties of Metals at Very Low Temperatures," ASM Preprint No. 3 (1956), *Transactions*, Am. Soc. for Metals, Vol. 49 (1957).

[3] E. T. Wessel, "Tension Testing Apparatus for the Temperature Range of −320 F to −452 F," ASTM BULLETIN, No. 211, Jan. 1956, p. 40 (TP 12).

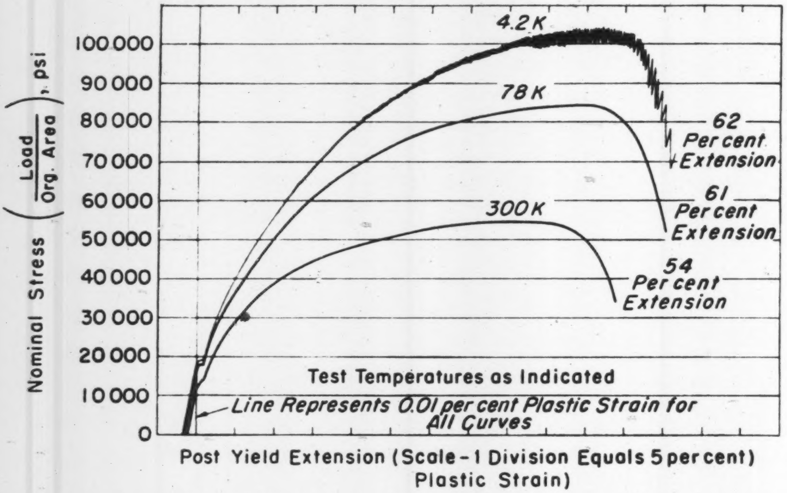

FIG. 16.—The Effect of Temperature on the Stress-Strain Curves of Commercially Pure Nickel (fcc) in the Annealed Condition.

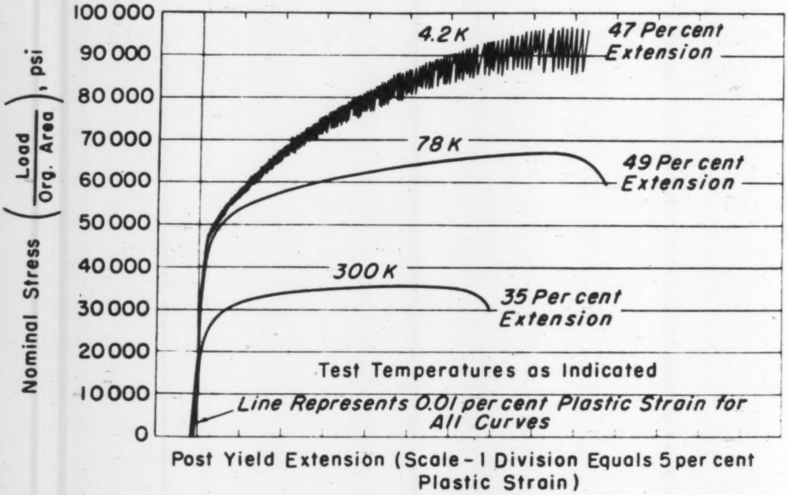

FIG. 17.—The Effect of Temperature on the Stress-Strain Curves of Crystal Bar, Vacuum Arc Remelted Zirconium (cph) in the Annealed Condition.

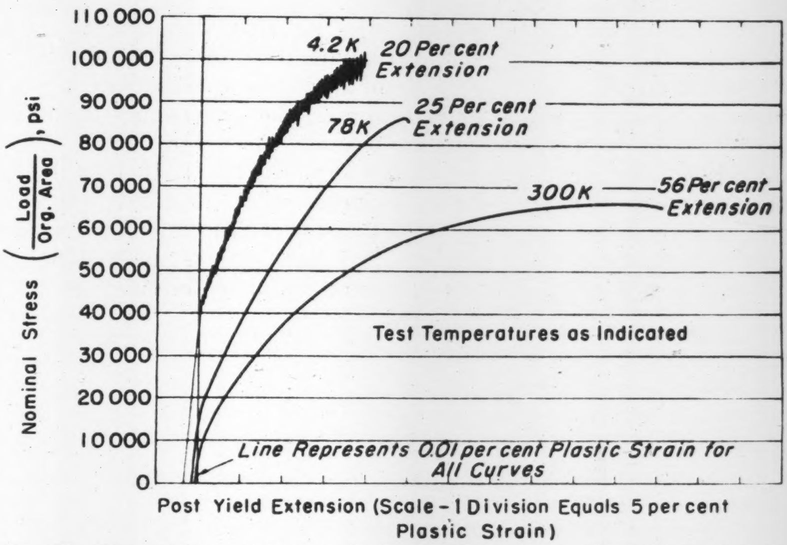

FIG. 18.—The Effect of Temperature on the Stress-Strain Curves of Relatively Pure Beta-Brass (bcc) in the Annealed Condition.

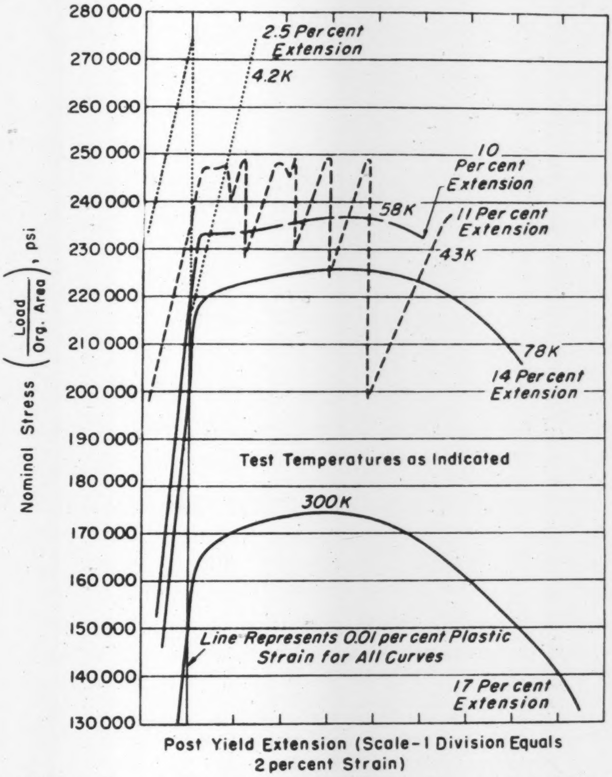

FIG. 19.—The Effect of Temperature on the Stress-Strain Curves of an Alloy Forging Steel (bcc) in the Quenched and Tempered Condition. (SAE 4340 Type)

in load and associated plastic strain increased while the frequency of the serrations decreased.

The onset of repeated yielding generally occurred in the temperature range of 35 to 45 Kelvin for most metals. As the test temperature decreased, the intensity of the repeated yielding increased, being most severe at 4.2 Kelvin. For those metals which became brittle at low temperatures, it was observed in many cases that the metal actually yielded and failed by brittle fracture during this initial yielding.[4]

The observation of repeated yielding as a mode of plastic deformation in so many metals of various lattice structures when tested at low temperatures suggests that one general mechanism may be responsible for the flow behavior of all metals at these very low temperatures. The exact mechanism for this behavior has not been conclusively determined but is thought to be associated with repeated mass release (bursts) of dislocations triggered by the high local stresses involved and aided to some extent by localized heating.

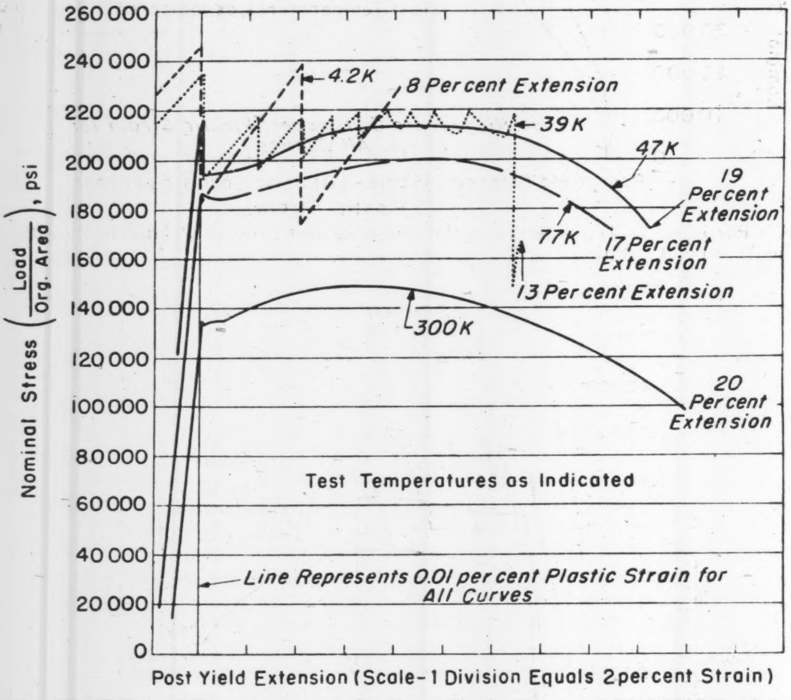

FIG. 20.—The Effect of Temperature on the Stress-Strain Curves of SAE 3335 Steel (bcc) in the Quenched and Tempered Condition.[5]

MR. GEORGE SACHS.[6]—It seems that some of the stainless steels, such as type 310, which possess a stable structure, did not develop discontinuous yielding, while other steels which have a tendency to form ferrite on straining show a pronounced jog. Is there any such relation between discontinuous plastic flow and transformations or precipitation of the alloy?

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[4] E. T. Wessel, "A Tensile Study of the Brittle Behavior of a Rimmed Structural Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 56, p. 540 (1956).

[5] R. D. Olleman, "A Tensile Study of Temper Brittleness in SAE 3335 Steel At Temperatures From 25 C to −269 C," Ph.D. Thesis, University of Pittsburgh, June 1955.

[6] Associate Director, Syracuse University Research Inst., Syracuse, N. Y.

MR. S. C. COLLINS (*author*).—It is beyond my competence to remark on this, though it may well be correct.

One thing should be pointed out which was not mentioned before. That is that these discontinuities are somewhat a function of the rate of strain, especially at the higher temperatures. For instance, in 310 and in 303, by going sufficiently slowly the onset of such things has been deferred until the temperature gets down low in the 20's—that is, 20 Kelvin or thereabouts—whereas if a reasonable speed is kept up they show up somewhere near 40 or even 45 on the test.

CHAIRMAN F. L. LAQUE[7].—Have the authors made any observation on possible changes in structure of the material as a result of a combination of low temperature and strain?

MR. COLLINS.—No, the examination has not been too professional. Photomicrographs were made, and some X-ray examinations. We found nothing.

At one time an assistant was very much interested in the matter of twinning, and found no correlation between that and the discontinuities.

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[7] Vice-president and Manager, Development and Research Div., The International Nickel Co., New York, N. Y.

THE EFFECTS OF STRAIN RATE AND TEMPERATURE ON THE STRESS-STRAIN RELATIONS OF DEEP-DRAWING STEEL*

BY R. J. MACDONALD,[1] R. L. CARLSON,[2] AND W. T. LANKFORD[3]

SYNOPSIS

This paper discusses the effects produced by a range of strain rates (0.002 to 0.8 in. per in. per sec) and temperatures (75 F. to 300 F) on the stress-strain properties of fully aluminum-killed temper-rolled steel. It is shown that the yield point phenomena and work-hardening characteristics of steel can be markedly affected by variations in both strain rate and temperature. Reference is made to dislocation theory and to velocity-modified temperature relations. Strain hardening is described by the exponent n (from the equation $\ln \sigma = \ln B + n \ln \delta$), and also by the tangent modulus. It is concluded that the latter index offers a more reliable indication of strain hardening.

The ability of a sheet metal to stretch uniformly over relatively large areas without a localization of deformation during deep-drawing and stretching operations is a highly desirable characteristic. Although a simple tension test does not completely simulate the complex conditions existing during such operations, wide experience has provided some insight into tensile characteristics that are desirable. In general, these characteristics are related to the type of flow observed—homogeneous or discontinuous—and the rate of strain hardening. The behavior of a given material, however, can vary considerably over wide ranges of strain rate and temperature. To realize its true potential, therefore, a material should be studied over whatever ranges of these variables are representative of actual practice.

With conventional tension testing equipment, for example, the maximum rate of head movement is in the neighborhood of 1.0 in. per min. During deep drawing, however, rates approaching 100 in. per min are common. To simulate, as nearly as possible, the deformation rates encountered during deep-drawing operations, special tension testing equipment was designed for use in this investigation. This paper presents data on the effects of strain rate and temperature on the stress-strain properties of deep-drawing steel.

EXPERIMENTAL PROCEDURE AND MATERIAL

High strain rates were obtained by using a Denison hydraulic press in conjunction with a specially designed sub-

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] Principal Metallurgist, Battelle Memorial Inst., Columbus, Ohio: now Senior Metallurgist, Clevite Research Center, Cleveland, Ohio.

[2] Assistant Division Chief, Battelle Memorial Inst., Columbus, Ohio.

[3] Chief Research Engineer, Specialty Products, United States Steel Corp., Monroeville, Pa.

press.[4] The Denison hydraulic press had a maximum load capacity of 10 tons and could be adjusted so that the rate of ram movement could be varied from about $\frac{1}{4}$ in. per min to 190 in. per min. Using a standard sheet tension specimen with a 4-in. gage section, strain rates from 0.001 to 0.8 in. per in. per sec were possible.

To transfer the downward movement of the hydraulic press to that of a tensile force on a sheet-steel specimen, a jig or subpress was designed. This subpress is shown in the schematic drawing in Fig. 1. The subpress consists of an upper stationary head, *A*, supported by four posts, *B*, and a lower movable head, *C*, from which two rods, *D*, extend upward through bearing-fitted holes in the stationary head, and contact the moving ram of the press. A dynamometer or load-measuring bar, *E*, is attached to the upper head, *A*, in series with the upper specimen grip, *F*. The lower specimen grip, *G*, is attached directly to the lower head, *C*. To adapt the subpress for tests at elevated temperatures, an insulated tank was attached to the lower movable head. This arrangement allowed the specimen to be completely submerged in heated oil during the test. Oil temperatures were maintained constant within $\pm\frac{1}{2}$ F by a thermostatically controlled knife heater inserted into the tank.

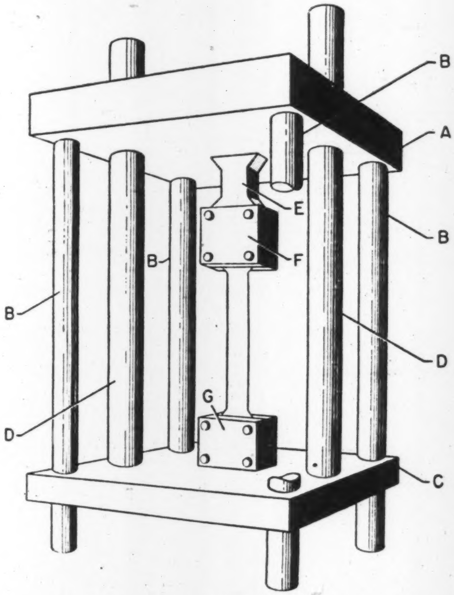

FIG. 1.—Subpress for High-Speed Tension Tests.

Head speeds were measured by timing electronically the rate at which the moving ram of the press traveled a calibrated distance during the tension test. The tensile load was measured by the dynamometer bar linked with the stationary head of the subpress. SR-4 wire strain gages were mounted to two opposite faces of the bar. The strains produced in the dynamometer bar with the application of a tensile load were measured and recorded by a Brush strain analyzer and oscillograph. The dynamometer bar was calibrated in a Baldwin-Southwark universal testing machine.

Strain during the tension test was measured by an extensometer, which was a special adaptation of the clip gage. It was so designed that the clip gages could be located outside of the constant-temperature bath. With this arrangement, the extensometer was fully extended when the knife-edged clamps were set at the 2-in. gage length. As the specimen elongated, the clip gages were allowed to return to their normal relaxed position. This permitted the test to be continued to failure without the necessity of removing the extensometer. SR-4 wire strain gages were mounted to each face of the extensometer loops. The signal from the strain gages was amplified and recorded on the two-channel Brush oscillograph, simultaneously with the applied load. A typical load-extension pattern, as recorded by the Brush oscillograph, is shown in Fig. 2.

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[4] This equipment is described in greater detail in a paper by MacDonald, *et al.*, entitled "An Apparatus for the Determination of Stress-Strain Properties at High Rates of Strain" to be published in the Proceedings of the Society for Experimental Stress Analysis, Volume XIV, Number 1, 1956.

True stress - true strain data were obtained from nominal stress and strain values by the use of the following relations:[5]

$$\sigma = \frac{L}{A_o}(1 + \epsilon),$$

$$\delta = \ln(1 + \epsilon),$$

in which:

σ = true stress, psi,

δ = true strain, in. per in.

L = applied load, lb,

A_o = original cross-sectional area, sq in., and

ϵ = nominal strain, in. per in.

Using calculated values of true stress and true strain, ln *versus* ln plots were made, and the data were found to satisfy the well-known empirical relation $\sigma = B\delta^n$. The constants B and n were obtained by graphical measurement.

Data concerning initial yielding behavior, that is, the existence of a yield point phenomenon, were obtained from the recorded values of load and nominal strain indicated by the Brush oscillograph. True stress and true strain values at maximum load were determined by observing the point at which the recorded value of applied load became constant. In many instances, however, this measurement was difficult because of the flatness of the load curve in this region. Such a condition, therefore, did not permit as accurate readings of true strain at maximum load as was desired.

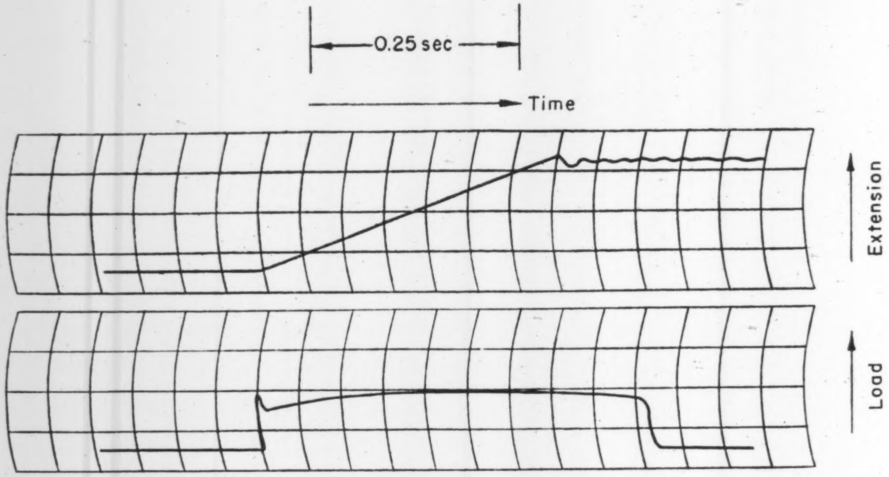

Fig. 2.—A Typical Load-Extension Curve.

The material used in this investigation was a fully aluminum-killed, temper-rolled sheet steel which was supplied by the United States Steel Corp. The average thickness of this sheet stock was 0.038 in.

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[5] In obtaining these relations it is assumed that the volume remains constant during straining.

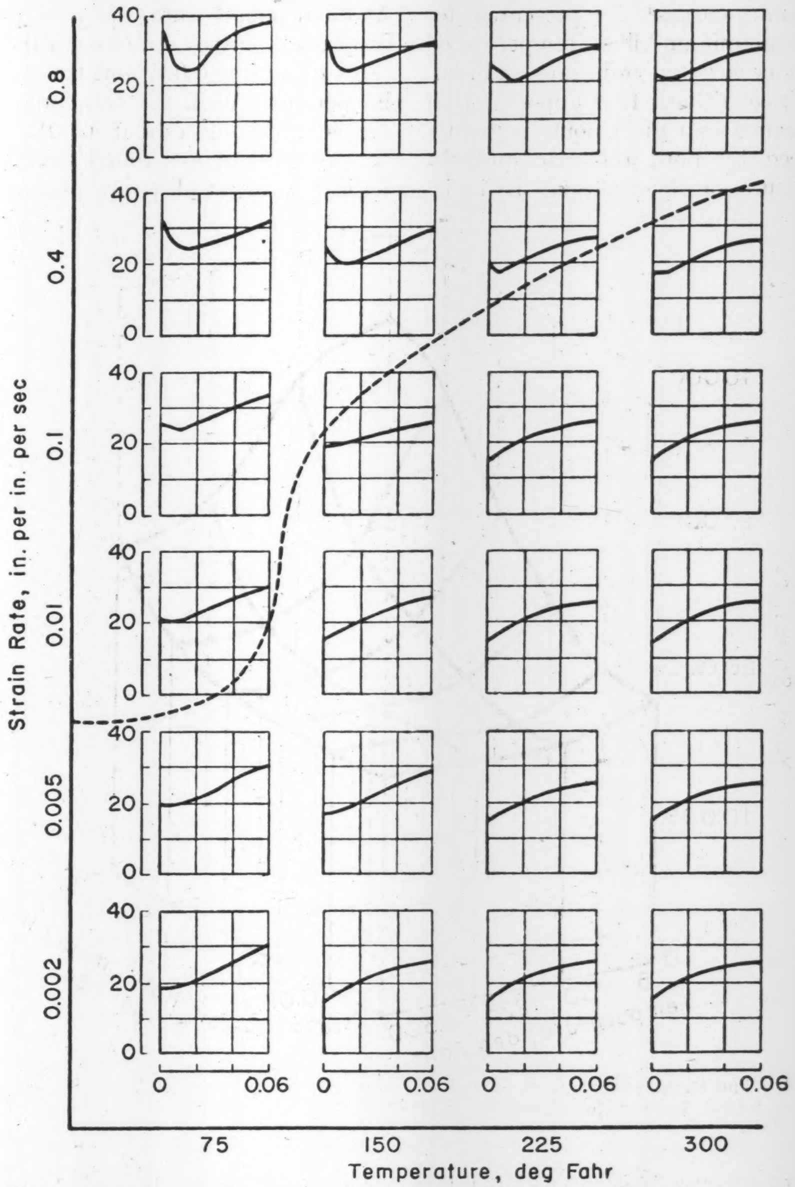

FIG. 3.—Nominal Stress-Strain Curves for Special-Killed, Temper-Rolled Steel as Affected by Strain Rate and Temperature.

TEST RESULTS

In this investigation, the effects of strain rates of 0.002, 0.005, 0.01, 0.1, 0.4, and 0.8 in. per in. per sec, and temperatures of 75 F, 150 F, 225 F and 300 F were studied. The data were analyzed with special attention given to the initial yielding behavior and the work-hardening index.

In Fig. 3, a series of typical nominal

stress-strain curves[6] are presented for the fully aluminum-killed, temper-rolled steel under varying strain-rate and temperature conditions. It is apparent that the presence of a yield point is greatly influenced by both the rate and the temperature of deformation. It is interesting to note that a yield point is evident with temper-rolled steel even at the highest temperature (300 F), if the rate of deformation is sufficiently large.

At room temperature, the yield point is suppressed only by straining at the lower rates of strain (0.005 and 0.002 in. per in. per sec). With the standard rate of tensile straining (about 0.001 in. per in. per sec), temper-rolled steel usually indicates no yield point phenomenon.

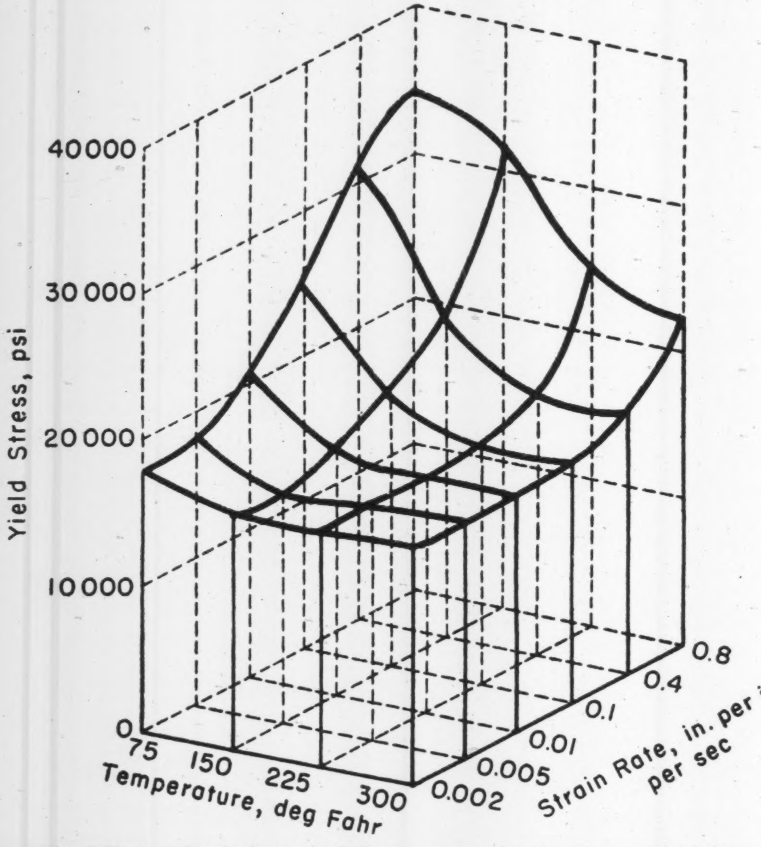

FIG. 4.—The Effect of Strain Rate and Temperature on the Upper Yield Stress of Fully Aluminum-Killed, Temper-Rolled Steel.

The dotted line in Fig. 3 designates approximately the strain-rate range above which a particular test temperature can result in a yield point. In observing the characteristics of the yield point, it is apparent that, for a given strain rate or temperature, there exists a gradual transition from a normal yielding be-

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[6] The results reported were taken from typical tests. Actually, a minimum of two specimens were used for each set of testing conditions. The agreement of data was within a deviation of 2 per cent.

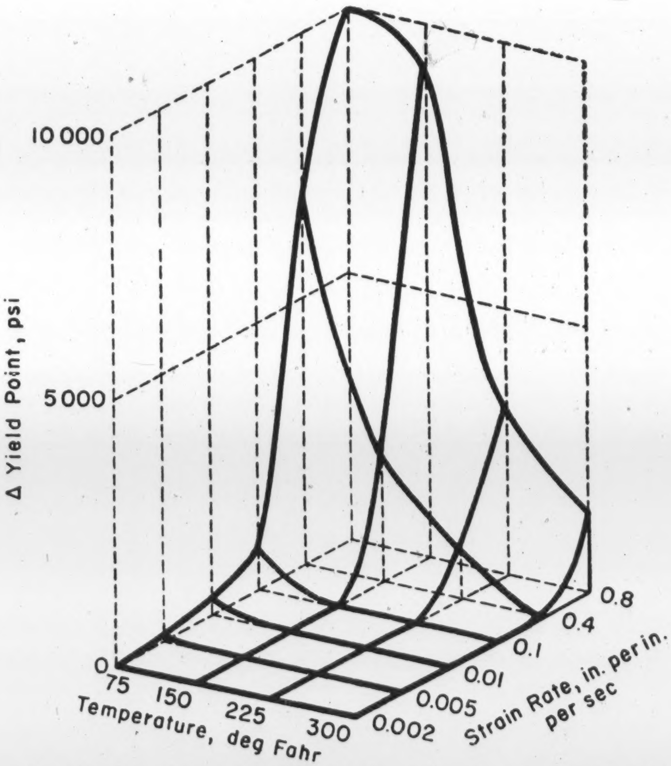

FIG. 5.—The Effect of Strain Rate and Temperature on the Difference Between Upper and Lower Yield Stresses of Fully Aluminum-Killed, Temper-Rolled Steel.

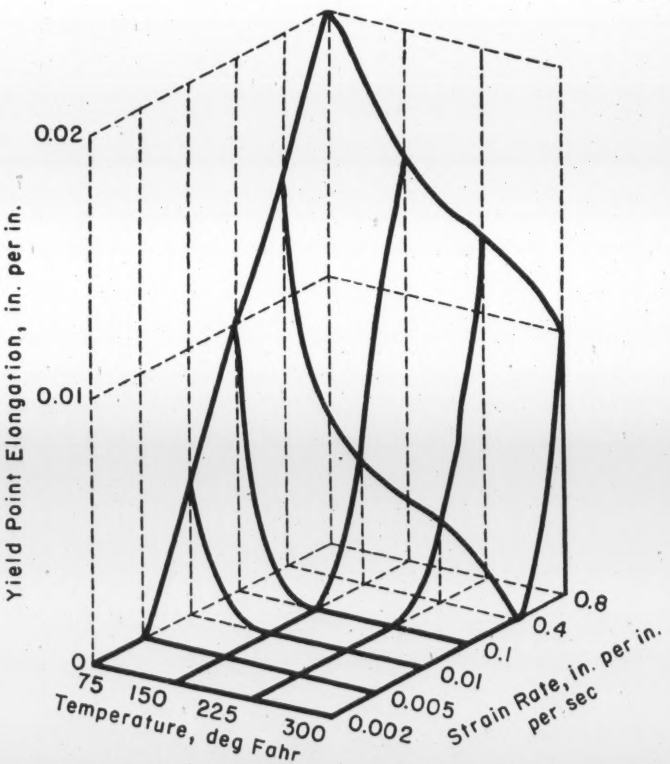

FIG. 6.—The Effect of Strain Rate and Temperature on the Yield-Point Elongation of Fully Aluminum-Killed, Temper-Rolled Steel.

havior (rounded stress-strain curve) to discontinuous yielding behavior.

In Figs. 4 to 9, the variations of stress-strain properties, as affected by strain rate and temperature, are represented graphically. The upper yield point (in Fig. 4) indicates a general tendency to increase with increasing strain rate and with decreasing temperature. As can be inferred from the stress-strain curves in Fig. 3, a significant increase in yield stress with variations in deformation conditions is apparent only when a yield point is indicated. When the yield point is suppressed, the variation in yield stress with either strain rate or temperature is negligible.

In Fig. 5, the term, Δ, yield point, represents the difference in stress level between the upper and lower yield points. It is significant that the intensity of yield point increases gradually with increasing rates of deformation and with decreasing temperatures.

The variation of the degree of nonhomogeneous yielding or yield point elongation associated with the yield point is shown in Fig. 6. The maximum amount of nonhomogeneous yielding (approximately 0.02 in. per in.) is indicated with a temperature of 75 F and a strain rate of 0.8 in. per in. per sec. It is interesting to observe that as the yield point becomes more intensive, the amount of nonhomogeneous yielding associated with it increases very rapidly.

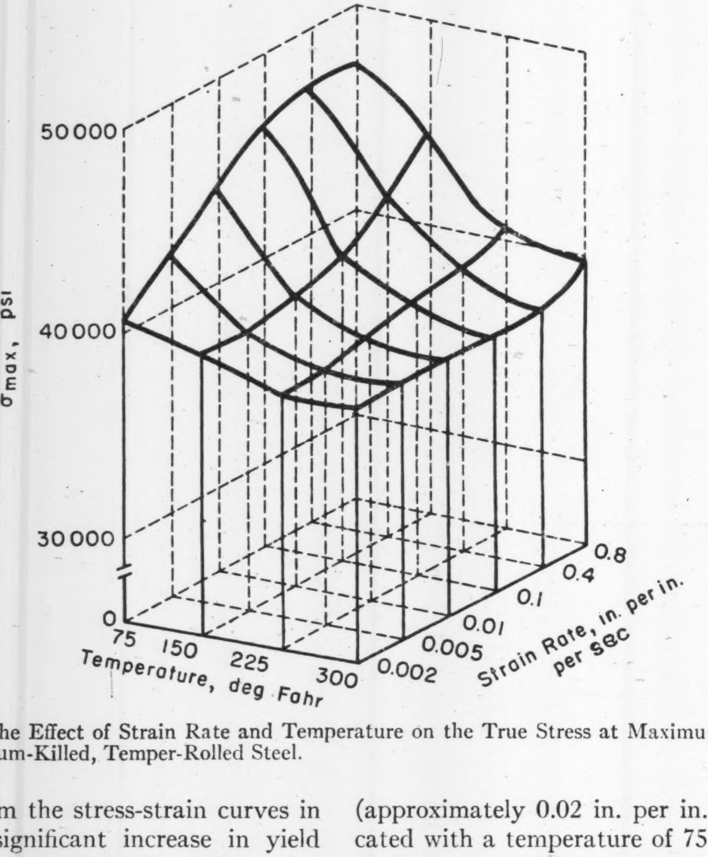

Fig. 7.—The Effect of Strain Rate and Temperature on the True Stress at Maximum Load of Fully Aluminum-Killed, Temper-Rolled Steel.

Figure 7 indicates the observed relations between strain rate, temperature, and the true stress at maximum load. In a comparison with the variations of initial yielding behavior, it can be noted that the value of true stress at maximum load is affected only under those conditions in which a yield point is exhibited.

In a similar fashion to the behavior of yield point phenomenon, the true stress at maximum load increases with greater rates of deformation and with lower temperatures.

Figures 8 and 9 indicate the dependencies of the constants n and B, from the empirical stress-strain equation $\sigma = B\delta^n$, with strain rate and temperature. With the fully aluminum-killed, temper-rolled steel used in this study, tension data fit a relation of the type $\ln \sigma = \ln B + n \ln \delta$.

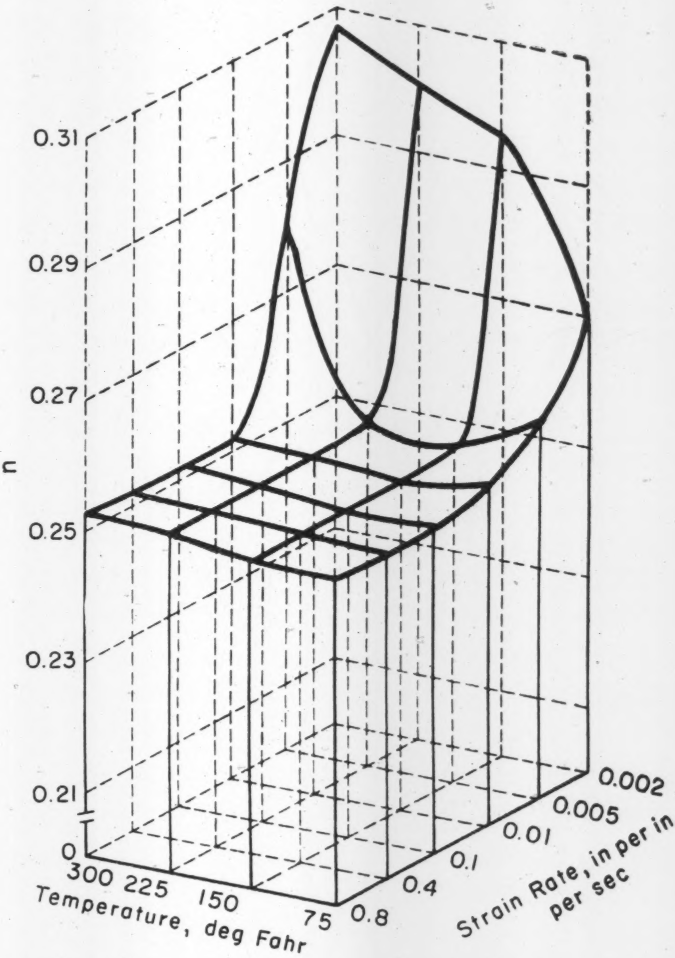

FIG. 8.—Effect of Strain Rate and Temperature on the Work-Hardening Index, n, of Fully Aluminum-Killed, Temper-Rolled Steel.

Essentially, these data for the variation of n with B with strain rate and temperature suggest that these parameters become sensitive to change only in certain strain-rate and temperature ranges. Increases in both *n* and *B* seem to be apparent with the lower strain rates and with the higher test temperatures. With lower test temperatures and greater strain rates, *n* remains essentially constant, although *B* exhibits a tendency toward rate and temperature sensitivity much like that reported for the upper yield stress and the true stress at maximum load. The significance of these findings will be discussed in a later section of this paper.

The true strain measured at maximum load generally reveals no significant variation with either strain rate or temperature.

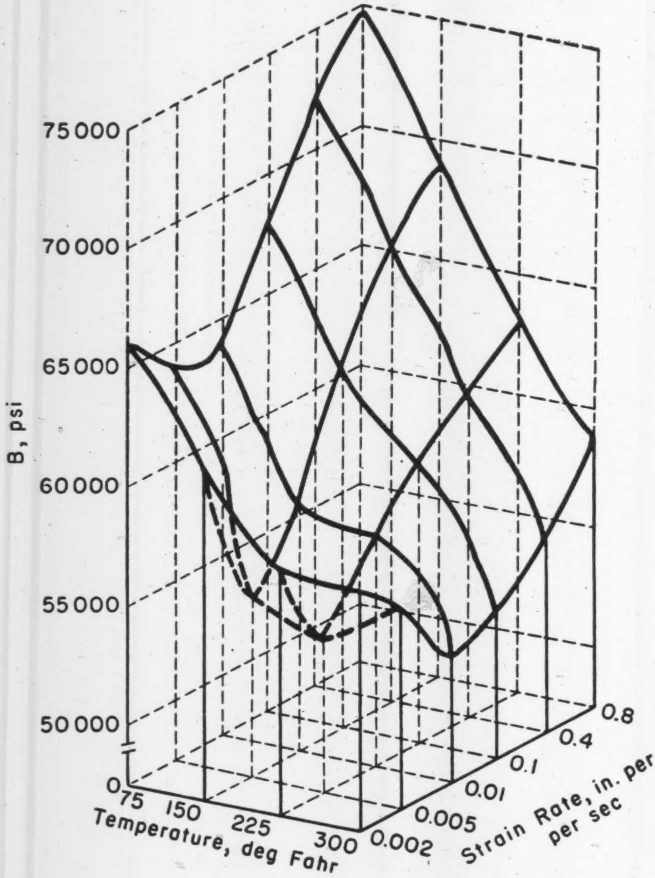

Fig. 9.—Effect of Strain Rate and Temperature on the Strength Index, *B*, of Fully Aluminum-Killed, Temper-Rolled Steel.

Discussion of Results

The results of this study have indicated that both strain rate and temperature can markedly alter the general appearance of the stress-strain curve of deep-drawing steel. The discussion of the relations between straining conditions and stress-strain properties will be divided into four sections: (1) initial yielding behavior, (2) factors influencing flow, (3) indices of strain hardening, and (4) application of equations of state.

Initial Yielding Behavior:

The yield point phenomenon commonly observed with low-carbon steel can be suppressed under straining conditions by slightly working the steel prior to deep drawing or stretching. This is usually accomplished by temper rolling with extensions of from ½ to 2 per cent. The suppression of the region of nonhomogeneous yielding and the lowering of the upper yield stress by temper rolling has been suggested by Polakowski (**1**)[7] and Hundy (**2**) to be related to the plastic working of the surface layer during the reduction operation. They attribute this effect to the residual stress distribution in the metal sheet (tensile stresses at the worked surface and compressive stresses at the center). Because of the existence of tensile stresses in the surface layer, yielding will be initiated at a lower stress value than that which would be required in the absence of residual stresses. Any tendency, however, for the surface grains to yield in a cataclysmic manner, as typified by the yield point phenomenon, will be restrained by the still elastic core of the sheet. The over-all result is a gradual slope or rounding of the stress-strain curve.

In this investigation, it was found that the characteristic yield point associated with annealed steel can be suppressed by temper rolling only for certain test conditions. In general, high strain rates or low test temperatures still allowed the formation of a yield point, which ordinarily would not be exhibited by temper-rolled steel under normal testing conditions. The fact that a yield point exists under certain conditions of deformation suggests that temper rolling only serves to decrease the rate and temperature sensitivity of the steel. In light of this observation, the removal of the yield point according to the "residual stress" explanation probably requires modification. It may be possible, for example, that a "size effect" must be accounted for.

As theorized by Cottrell and Bilby (**3, 4**), the yield point phenomenon observed with annealed steel may be associated with the collection of "atmospheres" or "clouds" of solute atoms around dislocations. These solute atmospheres are thought to be composed of carbon or nitrogen or both carbon and nitrogen, atoms. It is postulated that, in order to tear dislocations away from their restraining atmospheres, a high applied stress is necessary. Once these dislocations are released from these barriers, however, they can travel through the specimen at a reduced load. Thus, the sharp yield point commonly found with annealed, low-carbon steels can apparently be explained by the use of the theory of dislocations.

A few investigators have attempted to relate the combined effects of strain rate and temperature by a single mathematical expression, such as the use of a velocity-modified temperature. The test data collected from this study will be considered from this point of view in a later section of this paper.

FACTORS INFLUENCING FLOW

In discussing the dependencies of metal behavior upon strain rate and temperature during plastic deformation, it is useful to examine briefly the effects of dislocation and solute-atom movement. Once an atmosphere of solute atoms has collected around a stationary dislocation, any movement of the dislocation relative to the atmosphere results in a rise in strain energy. The solute-atom atmosphere thus exerts a restraining force on the dislocation. If the temperature and rate of deformation are such that the solute atoms can diffuse along with the moving dislocations, then the atmospheres can continue to exert a restraining force on the dislocation. For a given strain rate, therefore, there will be a temperature at which solute atoms are just sufficiently mobile to follow and to restrain the dislocations. The greatest strengthening effect should be apparent when the drift velocity of the solute atom equals the velocity of the dislocation itself. According to Cottrell (6), the condition for this is that the diffusion coefficient of the solute atom should be:

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[7] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 720.

$$D \approx (10^{-7}\ \text{sq cm})\,\dot{\epsilon} \ldots\ldots\ldots\ldots (1)$$

where:

$\dot{\epsilon}$ = strain rate per sec.

With the slowest strain rate considered in this study (0.002 in. per in. per sec), the diffusion coefficient from Eq 1 would be 10^{-10} sq cm per sec. According to diffusion data established for both carbon and nitrogen in alpha iron at temperatures of interest in this study, it is found that the maximum diffusion coefficient (at 300 F) is of the order of 10^{-12} sq cm per sec. This is a deviation of about 100 from the theoretical value found with Eq 1. The theoretical diffusion rate, however, must be regarded as only a rough indication of the general order of magnitude. In experimental work reported by Cottrell (8), the smallest rate of diffusion of nitrogen at which strengthening occurred during plastic deformation also indicated a discrepancy of about 100 from the calculated value.

Although the agreement between experimental behavior and theory is off by a few orders of magnitude, the above explanation still is plausible for the observed increase in the work-hardening index at the lower strain rates and higher temperatures (see Fig. 8).

INDICES OF STRAIN HARDENING

No single laboratory test has yet been devised that gives completely adequate indices of the properties that determine the deep-drawing and stretching characteristics of a sheet metal. Despite limitations, however, existing tests and methods of analysis have served as useful guides for evaluating formability.

One of the most simple tests for studying plastic response is the tension test. In this study, as in many others, the tension data have been found to fit the well-known empirical relation $\sigma = B\delta^n$. In this expression, the constant n is usually referred to as the strain-hardening exponent. Since this designation implies a sensitivity to strain hardening, it seems reasonable to regard the constant as an index of strain hardening. The *actual* rate of strain hardening, however, can be obtained by differentiating the equation $\sigma = B\delta^n$ to obtain the tangent modulus:

$$\frac{d\sigma}{d\delta} = Bn\delta^{n-1} \ldots\ldots\ldots\ldots (2)$$

For purposes of comparison, consider the value of this expression at the true strain at maximum load. Since the true strain at maximum load is mathematically equal to the constant n,

$$\left.\frac{d\sigma}{d\delta}\right|_{\delta=n} = Bn(n)^{n-1} \ldots\ldots\ldots\ldots (2)$$

or

$$\left.\frac{d\sigma}{d\delta}\right|_{\delta=n} = Bn^{n} \ldots\ldots\ldots\ldots (3)$$

Using this expression, tangent-modulus calculations at maximum load were made for the test data obtained in this study. The results, together with the corresponding values of n and B are

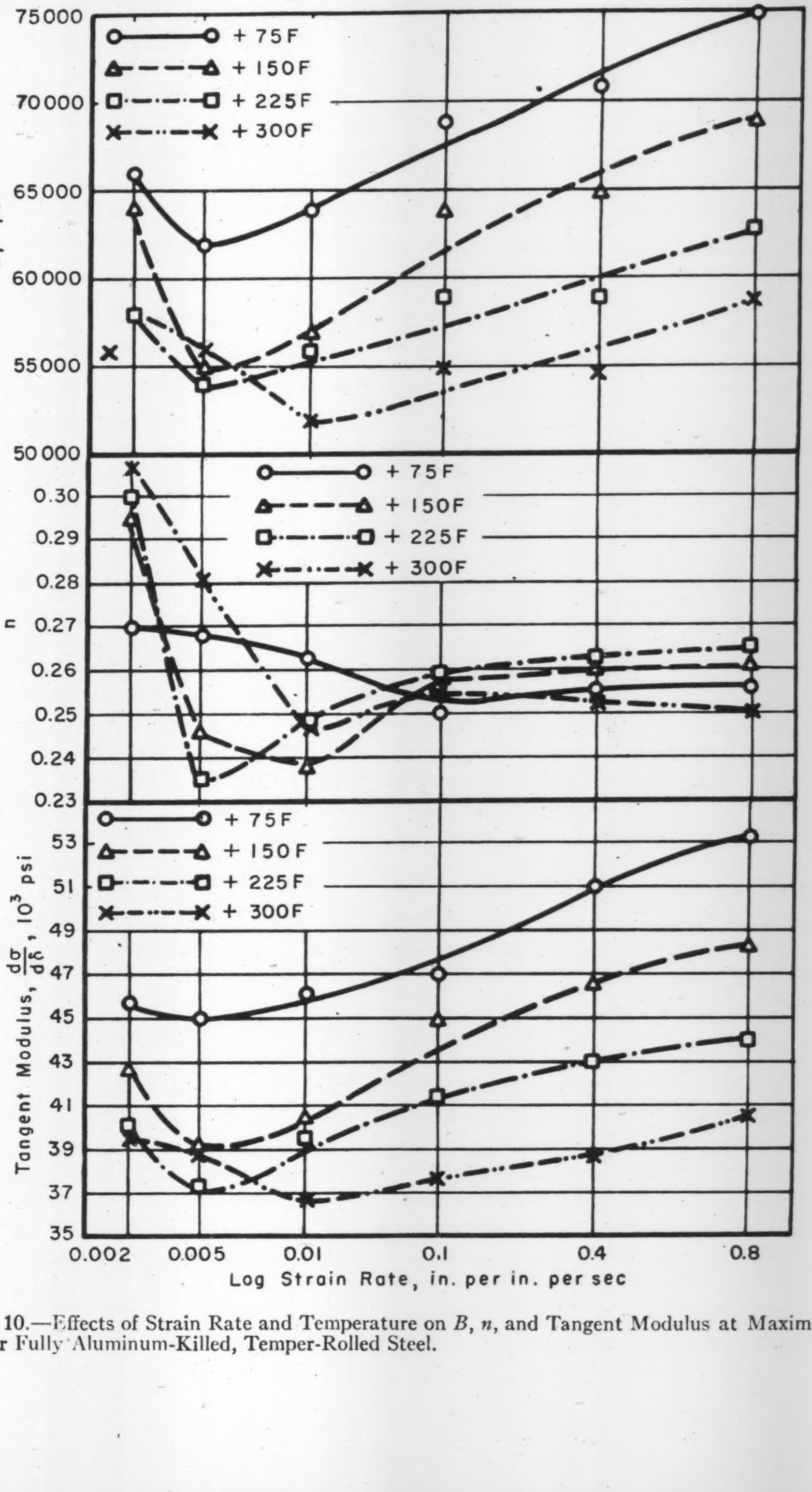

FIG. 10.—Effects of Strain Rate and Temperature on *B*, *n*, and Tangent Modulus at Maximum Load for Fully Aluminum-Killed, Temper-Rolled Steel.

given in Fig. 10. Comparisons of the corresponding curves indicate that, in general, the trends of the variations for $\frac{d\sigma}{d\delta}$ and B are similar. The variation of n, however, is dissimilar to that for $\frac{d\sigma}{d\delta}$ and B. Whereas the plot of n *versus* strain rate indicates that n is relatively constant for rates greater than 0.10 in. per in. per sec the tangent modulus shows a steady increase with increasing strain rate.

The preceding discussion suggests that the constants n and B should be interpreted with caution. The constants undoubtedly are of value as indices of plastic behavior, and comparisons of n and B values for two metals will certainly provide useful information on the relative response to loading. Care, however, should be exercised in interpreting the significance of these constants. Reference to Fig. 10, for example, indicates that the strain-hardening exponent, n, is relatively unaffected by high strain rates. This is not an accurate picture of the strain-hardening response, however. The plot of the tangent modulus indicates the fallibility of such an interpretation. Here, a distinct sensitivity is observed, and it would appear that B, the so-called strength index, is a better strain-hardening index at maximum load than n.

APPLICATION OF EQUATIONS OF STATE

The search for support of mechanical equations of state has interested engineers for many years. Since such equations involve only instantaneous values of the variables, the "state" is independent of prior history. The gas laws are examples of valid equations of state. Experience has indicated, however, the equations of state for metals can be valid only over limited ranges of such variables as strain rate and temperature. Also, history has been observed to influence the final "state."

Several equations of state for metals have been based on the fundamentals of reaction-rate theory. One such approach has been suggested by MacGregor and Fisher (9) who formulated the concept of a velocity-modified temperature. According to their derivation, the stress for a particular value of strain was a function of a velocity-modified temperature, T_m. That is, the stress, S, could be described by the functional relation:

$$S = f(T_m) \quad (4)$$

where:

$$T_m = T\left(1 - k \ln \frac{\dot{\epsilon}}{\dot{\epsilon}_0}\right) \quad (5)$$

and k and $\dot{\epsilon}_o$ were related to reaction-rate equation constants.

MacGregor and Fisher found evidence that indicated that, so long as T_m remained fixed (T and $\dot{\epsilon}$ can be varied to maintain T_m constant), the value of S remained unchanged. This lends some support to the validity of Eq 4. It should be noted, however, that, although the range of temperatures studied experimentally was large (−298 to 1232 F), the range of strain rates was not too large (5×10^{-5} to 500×10^{-5} in. per in. per sec). Since the variation in strain rate studied in the program described by this paper is considerably greater (200×10^{-5} to $80{,}000 \times 10^{-5}$ in. per in. per sec), it is of interest to apply the velocity-modified-temperature concept to the data obtained.

To compute T_m, the values of k and $\dot{\epsilon}_o$ used by MacGregor and Fisher were utilized. According to their data, k was apparently the same for all of the metals they studied (annealed SAE 1020, annealed SAE 1045, and annealed brass). Since k is an index of strain-rate sensitivity, a lack of variation in k

for the different metals implies the same sensitivity to strain rate. There appears to be no reason to suspect, however, that *k* should not be different for different metals.

Plots of stress *versus* T_m for the fully aluminum-killed, temper-rolled steel are given in Fig. 11. The upper points indicate the stresses that correspond to a strain of 0.15 and the lower points those that correspond to a strain of 0.05. The groups of points are identified according to test temperature below the curves. For a given test temperature, the highest strain rate is the first point on the left of a given group.

The curves drawn in Fig. 11 indicate a fair correlation with the parameter T_m. Although the scatter appears to be significant, there does not seem to be a consistent trend. The randomness of the scatter, in fact, tends to suggest that the deviation of the points from the curves is of the order of the experimental accuracy. It should also be noted that changes in *k* did not materially reduce the scatter.

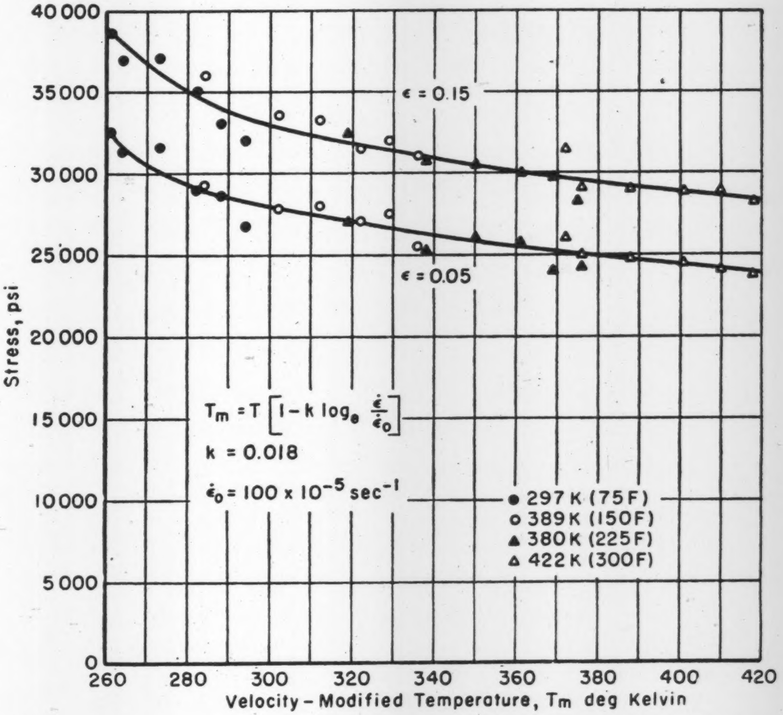

FIG. 11.—Stress *Versus* Velocity-Modified Temperature for a Fully Aluminum-Killed, Temper-Rolled Steel.

The stress *S* for each curve in Fig. 11 is described as the "stress reaction" caused by straining to a certain value of strain for a given T_m. If the upper yield stress can be considered the "stress reaction" at the initiation of plastic flow, it should be possible to observe a continuous variation of upper yield stress with T_m. To check this possibility, the plot of Fig. 12 was prepared. It is apparent from the plot that the upper yield strength is considerably more strain-rate sensitive than the remainder of the stress-strain curve. It is of interest to note, however, that, had tests been conducted only over rates of from 200 × 10^{-5} to 1000 × 10^{-5} in. per in. per sec, an apparent correlation, as indicated by the dashed curve, would have been obtained (the upper three points of each group would be missing).

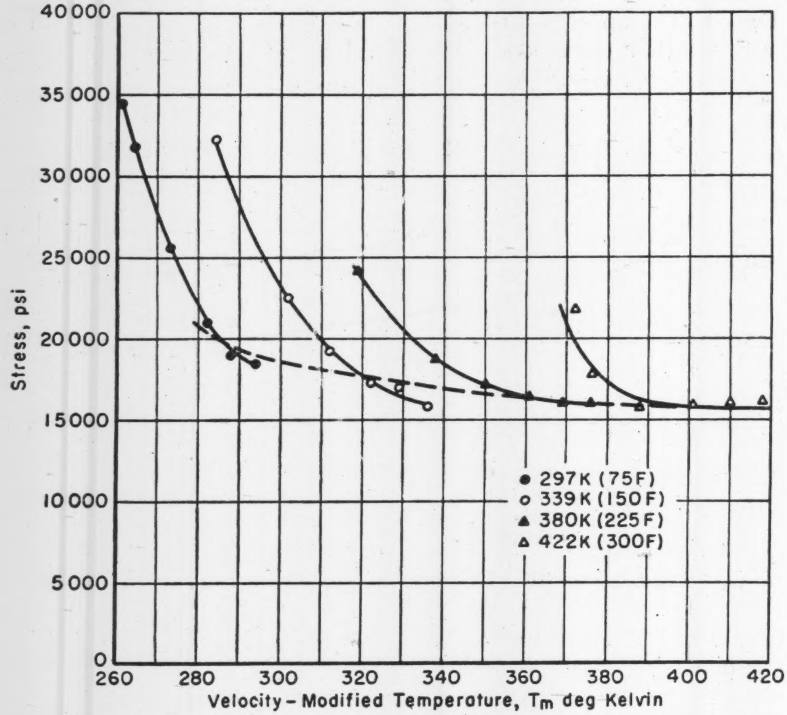

Fig. 12.—Upper Yield Stress *Versus* Velocity-Modified Temperature for a Fully Aluminum-Killed, Temper-Rolled Steel.

The nature of the deviation of the points for high strain rates from a continuous curve suggests that the validity of concepts such as the velocity-modified temperature could have what might be termed a "local validity." For a given value of the parameter T_m, for example, the acceptable values of $\dot{\epsilon}$ and T are bounded. If this is true, and it is reasonable, the equation $S = f(T_m)$ is not valid for all possible T_m.

A parameter of a form different from that described above has been suggested by Zener and Hollomon (10). This parameter is of the form:

$$P = \dot{\epsilon} \exp \frac{q}{RT} \qquad (6)$$

where:

$\dot{\epsilon}$ = the strain rate,
T = the absolute temperature,
R = the gas constant, and
q can be considered an activation energy.

In terms of stress reaction, S,

$$S = f(P)$$

If this equation is valid, constant values of P, for a given strain, will result in equal values of S.

Although the form of the above equation suggests a thermodynamic derivation, it apparently evolves from two empirical relationships. It has been found (11) that the relationship between strain rate $\dot{\epsilon}$ and stress, S, for a given strain and temperature is often described by

$$S = A(\dot{\epsilon})^n \qquad (7)$$

where:

A and n are constants. (NOTE.—A and n may vary with temperature.)

Further, it has been found that the relationship between stress, S, and absolute temperature for a given strain and strain rate can often be described by

$$S = B \exp \frac{Q}{RT} \qquad (8)$$

where:

B and Q are constants and R is the gas constant. (NOTE.—B and Q may vary with strain rate.)

If a functional relationship for the combined effects of strain rate and temperature exists, it should reduce to Eq 7 or Eq 8 when the temperature or strain rate is held constant. A functional form that satisfies this requirement has been suggested by Zener and Hollomon (9) of the form:

$$S = \mathrm{f}\left(\dot{\epsilon} \exp \frac{q}{RT}\right) \qquad (9)$$

Using the data obtained on fully aluminum-killed, temper-rolled steel for this program, it was found that Eqs 7 and 8 were satisfied. It was found, however, that n was temperature dependent and Q was strain-rate dependent.

Though the data were described by Eqs 7 and 8, Eq 9 was found to be invalid. The reason for the failure of Eq 9 can be seen by considering the behavior of n and Q.

Satisfaction of Eqs 7 and 8 implies:

$$S = K\left[(\dot{\epsilon})^n \exp \frac{Q}{RT}\right],$$

or

$$S = K\left[\dot{\epsilon} \exp \frac{Q}{nRT}\right]^n.$$

If $\frac{Q}{n} = q$, and q is constant, independent of temperature and strain rate, then:

$$S = K\left[\dot{\epsilon} \exp \frac{q}{RT}\right]^n \qquad (10)$$

Equation 10 can then be written in the functional form of Eq 9. That is:

$$S = \mathrm{f}\left(\dot{\epsilon} \exp \frac{q}{RT}\right).$$

Prior to Eq 10, however, it was noted that q must be constant *and* equal to $\frac{Q}{n}$. The value Q, however, was observed to be different for different strain rates, and n varied with temperature. In order, then, for q to be independent of temperature and strain rate, Q and n must vary in such a fashion as to maintain the ratio $\frac{Q}{n}$ constant. Since this condition was not satisfied, Eq. 9 was not valid for the data obtained. It is thus seen that data described by Eqs 7 and 8 are not necessarily described by the functional relationship indicated in Eq 9.

SUMMARY

This paper discusses the effects produced by a range of strain rates (0.002 in. per in. per sec to 0.8 in. per in. per sec) and temperatures (75 F to 300 F) on the stress-strain properties of fully aluminum-killed, temper-rolled steel. The equipment used for obtaining high-speed tension data is briefly described. In presenting the data, special attention is given to the yield point phenomena and the stain-hardening characteristics accompanying plastic deformation. It is shown that the yield point phenomena, which generally are absent with fully aluminum-killed, temper-rolled steel, occur with considerable intensity at high rates of strain. Reference is made to dislocation theory and to velocity modified-temperature relations. Strain-hardening is described by the exponent n (from the equation $\log \sigma = \log B + n \log \delta$) and also by the tangent modulus. It is concluded that the latter index offers a more reliable indication of strain-hardening.

Acknowledgement:

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DISCUSSION

MR. R. H. HEYER[1] (*presented in written form*).—The authors are indeed to be complimented on developing a new and promising method for obtaining data on the plastic stretching behavior of sheet metals and on their application of the method to aluminum killed deep drawing steel.

It has been known, from the work of Winlock and others, that high strain rates accentuate the yield point effect and increase the yield stress of rimmed low-carbon steels. It is also known that elevated testing temperatures have a marked effect on rimmed steels due to the so-called "blue-brittleness" phenomenon. The yield point phenomena developed by the authors on temper rolled aluminum-killed steels would not be predicted from these facts.

The authors state that the material tested is fully aluminum killed; however, it would be reassuring to know that the metallic aluminum content is 0.03 per cent or greater, since lower residual aluminum can result in marginal stability.

While the emphasis in the paper is on yield point behavior, it would be of interest to know more about the measurements of uniform elongation; that is, the strain at maximum load. The authors mention the difficulty of measuring this value and state that true strain at maximum load generally reveals no significant variation with either strain rate or temperature. This in itself is an interesting observation that the uniform elongation, which has often been advocated as a criterion of drawability, is insensitive to strain rate and temperature.

In devising Eq. 3 for tangent modulus the authors use the relationship "true strain at maximum load is mathematically equal to the constant *n*."

Did the authors use the true strain values represented by the constants *n* as a check against the true strain at maximum load as determined by observation of the curves?

Did the authors fit their calculated tangent modulus lines to the curves at the true strain values represented by *n* to observe the quality of the fit?

Admittedly our choices of drawing criteria are rather speculative at this point. The tangent modulus at maximum load now takes its place as one of the newest criteria.

MR. W. T. LANKFORD (*author*).—With reference to Mr. Heyer's question about the steel that was used, this steel would be considered as representative of good practice for fully aluminum-killed deep-drawing steel. The acid-soluble aluminum content was 0.065 per cent.

MR. R. J. MACDONALD (*author*).—In general the true strains observed at maximum load did not correlate with the measurements of the constant *n*. This may be interpreted in the following way.

Generally there was difficulty in measuring the true strain at maximum load because the recorded value of load on the oscillograph recording was quite flat, and therefore it was difficult to pick out the maximum point. The value of *n* was taken, however, as a convenient way of illustrating the use of the tangent modulus as an index of strain hardening.

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[1] Armco Steel Corp., Middleton, Ohio.

Future work will be directed along these lines and data fitting the tangent modulus curve will probably be reported in a future paper.

MR. J. J. PREISLER.[2]—Have the authors investigated the effect of amounts of temper rolling—that is, amounts of deformation in temper rolling—on the suppression of yield point, or lack thereof, at high strain rates?

MR. MACDONALD.—This is being done at the present time. Being no longer with Battelle, I am not familiar with this data at present. The investigation is under way, however.

MR. E. T. WESSEL.[3]—The authors are to be complimented on their paper, with particular reference to the extensiveness of the data and the straightforward manner in which it was presented. Some questions arise which, while not directly related to the primary purpose of this paper, are pertinent to the dislocation anchoring concepts (**3, 4**)[4] of the yield point phenomena as reviewed in this paper. These questions are as follows:

What minimum amount of non-elastic strain could be observed with the strain measuring techniques used in this investigation?

Were the authors able to observe any evidence of plastic strain prior to the upper yield point in those cases where a pronounced yield point was obtained? If so, what was the behavior of this pre-yield plastic strain as a function of both temperature and strain rate?

These questions are prompted by results of recent experiments[5] which indicate that appreciable amounts of non-elastic strain precede the upper yield point in many body-centered-cubic metals, particularly at low temperatures and slow strain rates. Such observations indicate that some modification of the dislocation anchoring concepts (**3,4**) of abrupt yielding is necessary since this mechanism does not necessitate any prior plastic strain of appreciable magnitude.

MR. MACDONALD.—In general I would not consider the strain measuring apparatus to be sensitive enough to pick up any strain measurements prior to the initial yielding. It is around 0.005 in. per in., but actually, the way a measurement is taken human error can be anticipated, inasmuch as it involves taking readings from the recorded graph.

Any information of this could not be type considered valid with such small strains.

MR. LANKFORD.—One of the most significant things that is shown up by this work is the fact—and, a lot of people have recognized this before but it is brought out a little more vividly here than previously—that one must be careful in saying that steel does or does not exhibit a yield point. One must say that it does or does not exhibit a definite yield point under certain specific conditions of strain rate and temperature.

In the other paper[6] that Mr. MacDonald mentioned, describing the testing technique in more detail, there is another example of the application of this technique in which the effects of variations in the strain rate and temperature on the stress-strain relationships for wet hydrogen-treated steel were studied.

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[2] Sperry Gyroscope Co., Division of Sperry Rand Corp., Engineering Department, Standards Engineering, Great Neck, N. Y.

[3] Research Engineer, Metallurgy Department, Westinghouse Electric Corp., Research Laboratories, Pittsburgh, Pa.

[4] The boldface numbers in parentheses refer to the list of references appended to the paper, see p. 720.

[5] Unpublished data—Westinghouse Research Laboratories, paper submitted to the American Institute of Mining, Metallurgical, and Petroleum Engineers, Inc.

[6] R. J. MacDonald, R. L. Carlson, and W. T. Lankford, "An Apparatus for the Determination of Stress-Strain Relationships at High Rates of Strain," *Proceedings*, Soc. Experimental Stress Analysis, Vol. 14, No. 1, pp. 163–170 (1956).

Several years ago Gensamer and Low showed that the yield point could be eliminated from either killed or rimmed steel by the use of a wet hydrogen treatment. If the treatment that they prescribed is carried out and the same strain rates are used at which they tested, there is no definite yield point. However, if higher strain rates or lower temperatures are used, there is a definite yield point with considerable amounts of yield point elongation.

MESSRS. R. L. CARLSON AND W. T. LANKFORD (*authors' closure*).—In the period since the presentation of this paper, some question has arisen with regard to the ability of the recorder used to reproduce faithfully the load-time response at the fastest strain rates. In these cases, attention would be focused on the interval during which the load is rising to the upper yield point. The recorder response is about 100 cps. For the fastest strain rate (0.8 sec^{-1}), an elastic modulus of 30×10^6 psi, and an upper yield stress of 30×10^3 psi, the time required to reach the upper yield would be of the order of 0.001 sec. This is clearly beyond the response of the recorder.

A check of the load records, however, revealed that the measured time (the chart speed was known) to the upper yield point for the fastest strain rate was of the order of 0.01 sec. Since the rise to the upper yield point constitutes, in essence, a half cycle, the load record should provide a faithful record of the load response.

Another point can be cited in support of the fact that the records obtained reproduce the actual conditions. The elastic modulus would not be expected to vary significantly in the temperature range studied (75 F to 300 F). The initial load impulse that the recorder would receive at a given strain rate should, therefore, be approximately the same. If the impulse time was less than that recordable, the record should be distorted in essentially the same manner for each of the test temperatures. Figure 3 of the paper, however, shows that a distinct change in characteristics occurs with increasing temperature even for the fastest rate. This suggests that the recorder was capable of responding to slight variations successfully.

The fact that the time to the upper yield point is greater than would be anticipated from the indicated strain rates, means that the strain rate in the elastic range was slower than that after yielding (the rate after yielding was very nearly constant and is given by the indicated values). The tabulation below gives the strain rate values prior to and after yielding for each of the strain rates used. The values were computed from the recorder records.

Approximate Strain Rate Prior to Yielding per sec	Strain Rate After Yielding per sec
0.1	0.8
0.05	0.4
0.01	0.1
0.002	0.01
0.0006	0.005
0.0003	0.002

In describing the yield point behavior, the governing strain rates are, then, those in the column at the left. The strain rate conditions beyond the yield point are given in the column to the right.

EFFECT OF GEOMETRY ON THE PROPERTIES OF C4A-T6 AND SG70A-T6 ALUMINUM-ALLOY CASTINGS*

By W. H. Johnson[1] and H. F. Bishop[2]

Synopsis

Tensile properties were determined at various locations in aluminum alloy (C4A-T6 and SG70A-T6) castings of different thicknesses and geometries and compared with properties obtained from separately cast test bars. The properties of C4A alloy castings were shown to be more sensitive to cooling rates than those of the SG70A alloy. The properties of C4A alloy castings are also affected to a greater extent by section thickness variations than in the case of SG70A alloy castings. It is shown that special molding and melting processes may be used to develop properties in castings which approach (alloy SG70A) or exceed (alloy C4A) test bar properties.

While separately cast test bars are used as a basis for accepting or rejecting castings made from a given heat, it is generally agreed that test bar properties are not fully representative of the properties of the castings. Test bars are designed, risered, and cast in a manner to produce optimum mechanical properties. The design features of saleable castings, however, are often such that conditions for obtaining maximum properties throughout the casting are not always present. Some specifications recognize the limitations of foundry practice with respect to aluminum alloys and require that test bars removed from castings shall have average tensile strengths and elongations which are not less than stated percentages of the values specified for separately cast test bars. Such downgrading of mechanical property requirements is necessarily somewhat arbitrary since there are very few published data pertaining to actual casting properties, but it does give the designer working constants which are more realistic than those indicated by separately cast test bars.

The present study was aimed at establishing relationships between test bar properties and properties which may be expected from aluminum alloy castings of different shapes and thicknesses. It was expected that such information would be of interest to the foundryman as well as the design engineer in that it may indicate preferable foundry processing procedures.

Experimental Procedure

Two aluminum alloys, C4A-T6 and SG70A-T6, were cast into the plate, L, T, and X shapes shown in Figs. 1 and 2. The smallest plate was 4 in. wide and 9 in. long; the second plate was enlarged

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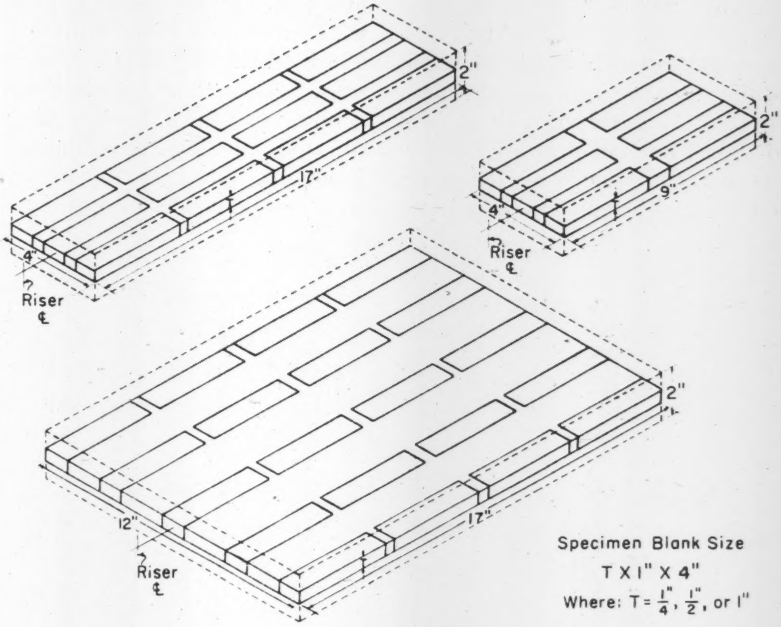
* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

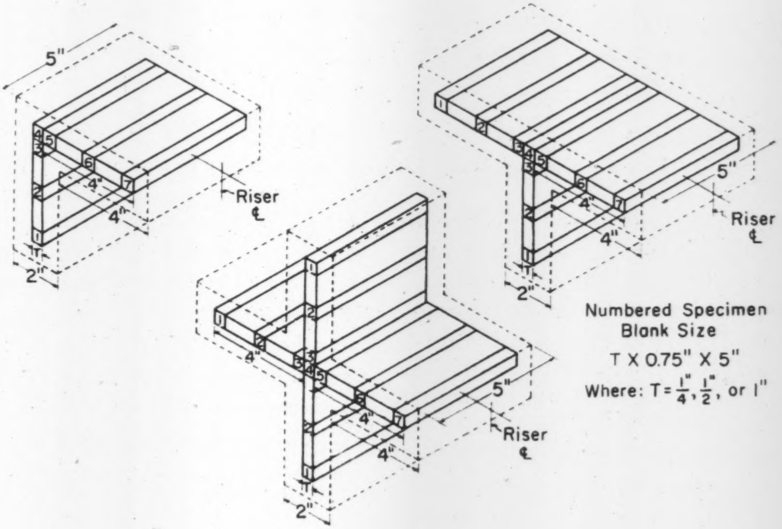
[1] Formerly, Head, Nonferrous Casting Unit, Naval Research Laboratory, Washington, D.C.; now Principal Metallurgist, Nonferrous Metallurgy Division, Battelle Memorial Institute, Columbus, Ohio.

[2] Metal Processing Branch, Metallurgy Div., Naval Research Lab., Washington, D.C.


2"
17"
4"
Riser
℄
2"
9"
4"
Riser
℄
2"
12"
17"
Riser
℄

Specimen Blank Size
T X 1" X 4"
Where: T = $\frac{1''}{4}$, $\frac{1''}{2}$, or 1"

FIG. 1.—Plate Castings.


5"
4
5
3
6
2
7
1
4"
4"
Riser
℄
2"
1
2
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7
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2"
1
2
1
2
3
4
5
6
7
4"
5"
4"
4"
Riser
℄
2"

Numbered Specimen
Blank Size
T X 0.75" X 5"
Where: T = $\frac{1''}{4}$, $\frac{1''}{2}$, or 1"

FIG. 2.—Joined Section Castings.

by increasing the length to 17 in.; the largest plate was maintained at the 17-in. length but increased in width to 12 in. Each of the 6 shapes were cast in thicknesses of $\frac{1}{4}$, $\frac{1}{2}$, 1, and 2 in. with each of the two alloys. Each of the various castings was fed with one 4-in. diameter, 9-in. high riser with the metal gated into the riser. The risers were attached to the casting edges at the positions indicated in Figs. 1 and 2. No chills were employed in this investigation. Pouring temperatures were 1250 F except for the $\frac{1}{4}$-in. thick castings which were poured at 1350 F. The purpose of the increased plate dimensions was to determine how mechanical properties within castings are affected when the feeding range required of a riser is increased, and also to evaluate the influence of edge effects, which are known from studies with other alloys (**1, 2**)[3] to influence casting soundness. The L, T, and X sections simulate conditions which would exist in complicated castings containing section junctions. Actually, if these sections were to be made as integral castings, better feeding and better properties would undoubtedly be obtained with risers located at the heavy section formed by the junction of the component members; however, when such junctions are part of complex castings, it is often impossible to do this.

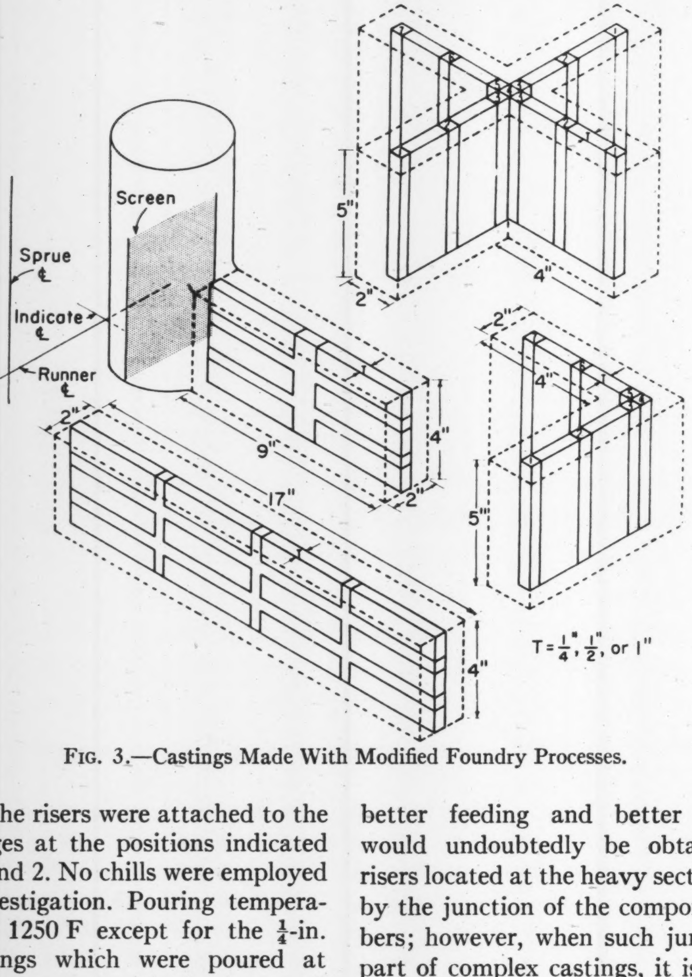

FIG. 3.—Castings Made With Modified Foundry Processes.

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 736.

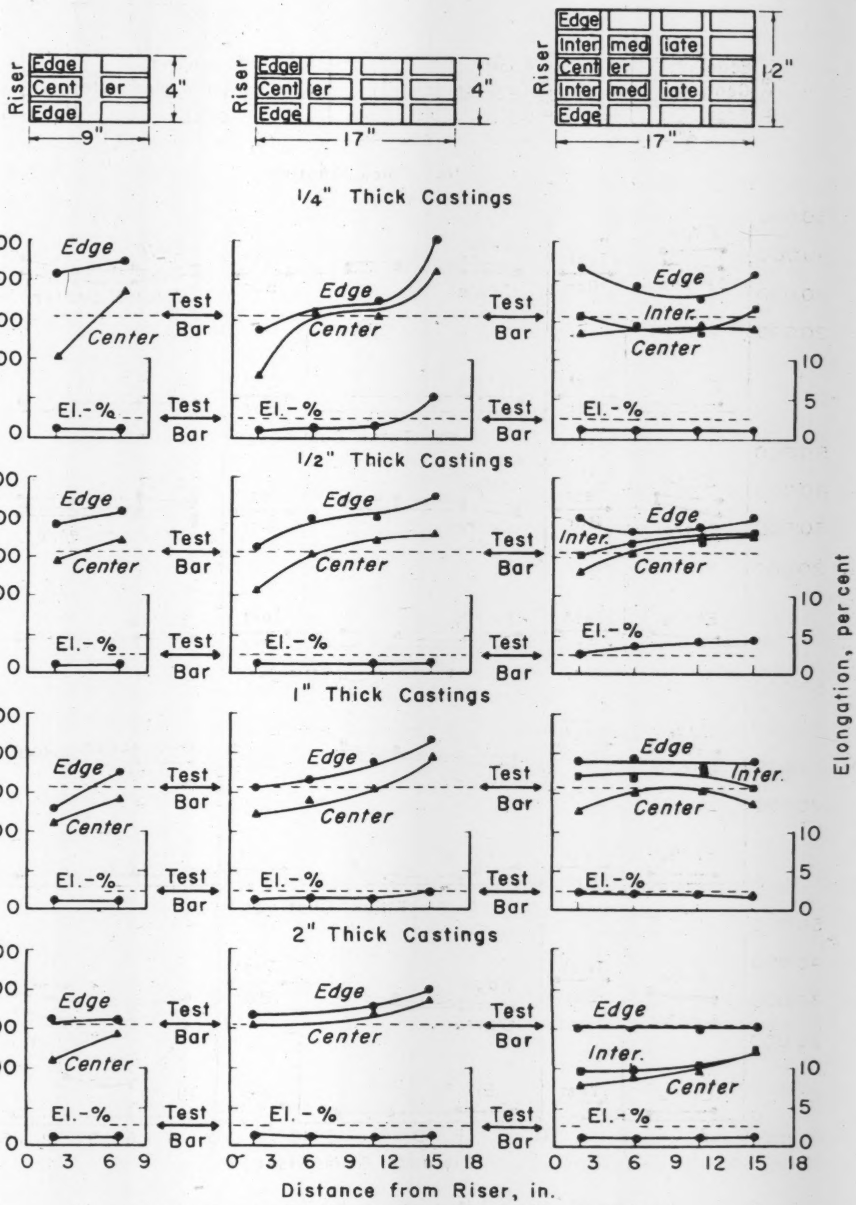

FIG. 4.—Mechanical Properties in C4A-T6 Plate Castings.

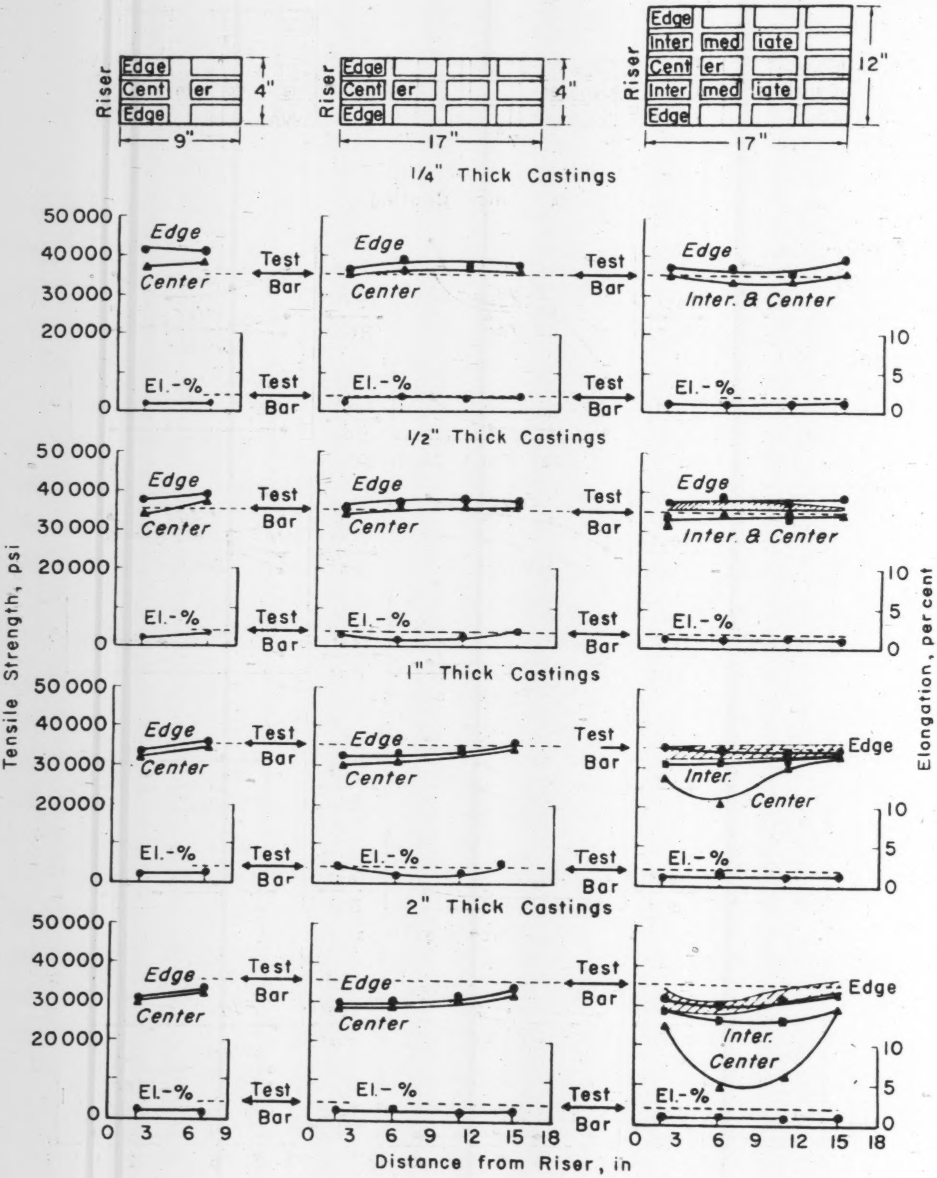

FIG. 5.—Mechanical Properties in SG70A-T6 Plate Castings.
Shaded areas refer to castings made with insulated risers.

Standard $\frac{5}{16}$-in. web Webbert test bar castings were poured with each heat and heat treated along with the castings so that comparison of properties could be made. After heat treatment, test bar blanks were machined from the castings at locations indicated in Figs. 1 and 2. Standard 0.505-in. diameter shoulder grip specimens were machined from the web bar and from the 1- and 2-in. sections; 0.357-in. diameter specimens were obtained from the $\frac{1}{2}$-in. thick castings and sheet specimens, $\frac{1}{4}$ by $\frac{1}{2}$ in. in cross-section, were obtained from the $\frac{1}{4}$-in. thick castings.

TABLE I.—COMPARISON OF TENSILE PROPERTIES OBTAINED FROM THREE TYPES OF TEST SPECIMENS OF ALLOY C4A - T6.

Specimen Type	Tensile Strength, psi	Elongation, per cent
0.505 D.	34 400 31 200 34 000	2½ 2 2½
Average	33 200	2½
0.357 D.	33 800 33 000 33 900	2½ 2½ 2½
Average	33 600	2½
¼ × ½.	32 500 29 500 33 900	2½ 3 2½
Average	32 000	2½

Only one of each type of casting was made by the procedures illustrated in Figs. 1 and 2. However, a few of the test castings were remade using different molding and melt treatment techniques. These castings, Fig. 3, were made such that all the casting sections were in the vertical position rather than the horizontal position and were gated by means of the American Foundrymen's Society recommended (3) rectangular tapered sprue and runner system for light metals. In addition, a strainer screen was incorporated in the riser so that any possible dross would be removed from the metal before it entered the casting. The melts in these latter tests were treated with a grain refiner after the degassing treatment. This consisted of stirring 0.20 per cent titanium, as a 6 per cent titanium-aluminum alloy, into the melt. The alloys were melted in an oil-fired furnace using charges of aluminum ingot and either copper-aluminum or silicon-aluminum master alloys. The magnesium in the SG70A alloy was added as commercially pure magnesium. The C4A alloy castings were poured from seven heats containing 4.24 to 4.58 per cent copper; the SG70A alloy castings were poured from seven heats containing 6.66 to 6.95 per cent silicon and 0.20 to 0.27 per cent magnesium. The total amounts of residual elements in these alloys, except in the cases where grain refiners were added, was below 0.35 per cent.

Data Obtained

Table I shows comparisons of tensile properties obtained with the three test bar designs employed to determine casting properties. The properties in Table I were obtained from specimens removed from the same locations in keel block castings poured from a single heat. It may be noted that the properties indicated by the 0.357-in. and 0.505-in. diameter specimens are in close agreement while the flat specimens exhibit lower tensile strengths. There is no alternative to the use of the flat specimens as a means of determining properties in the $\frac{1}{4}$-in. thick castings, and the data reported for these cases may be weighted according to the indications of Table I.

Figures 4 and 5 show properties obtained from various locations in the horizontally cast plates made with the two alloys and are compared with the average properties obtained from the sep-

TABLE II.—TEST BAR PROPERTIES.

Alloy C4A-T6				Alloy SG70A-T6			
		Modified Practice				Modified Practice	
Tensile Strength, psi	Elongation, per cent	Tensile Strength, psi	Elongation, per cent	Tensile Strength, psi	Elongation, per cent	Tensile Strength, psi	Elongation, per cent
33 000	3	34 700	3	38 000	3	30 400	5
33 000	3	36 500	3	40 000	2½	31 100	5
30 000	3	34 500	3	35 400	2	28 900	5
31 000	4	35 900	3	33 600	3½	31 500	3½
30 700	3	34 500	3	35 000	2	30 750	4
31 700	4	38 700	4	34 000	2½	30 000	4½
31 500	3	37 750	5½	33 000	3½	33 150	4
		38 250	6	33 500	2	31 200	3½
		36 500	5	33 000	2		
				35 700	4		
				34 500	4		
Average 31 500	3	36 400	4	35 000	3	30 900	4½

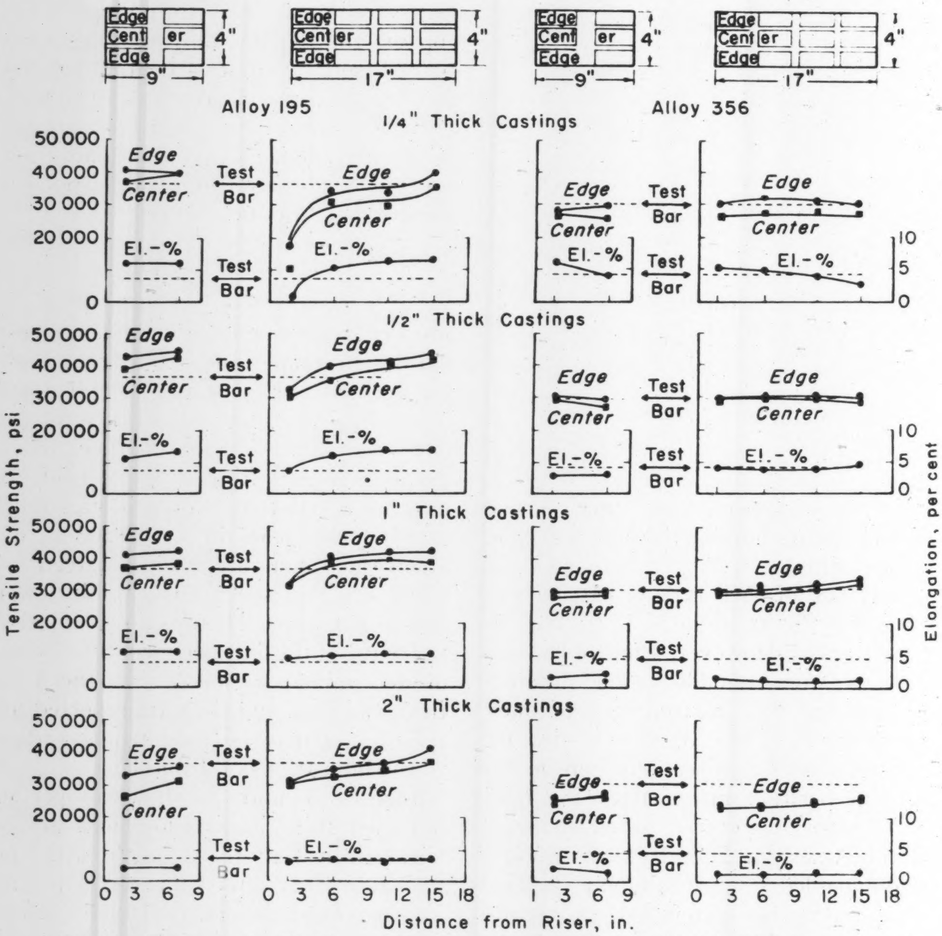

FIG. 6.—Properties in Aluminum Alloy Plate Castings Made With Modified Processes.

arately cast test bars, Table II. The properties obtained at the two edges in each plate and at the two intermediate locations in the largest plate are shown as averages in the figures since in effect they are duplicate tests and the properties were in close agreement. Tensile strengths in the C4A alloy plates are relatively sensitive to cooling rates. It may be noted (Fig. 4) that strength values are higher at the faster cooling plate extremities and edges than they are at the slower cooling center regions and locations adjacent to the riser. Conversely, the SG70A alloy, Fig. 5, is relatively insensitive to cooling rate variables

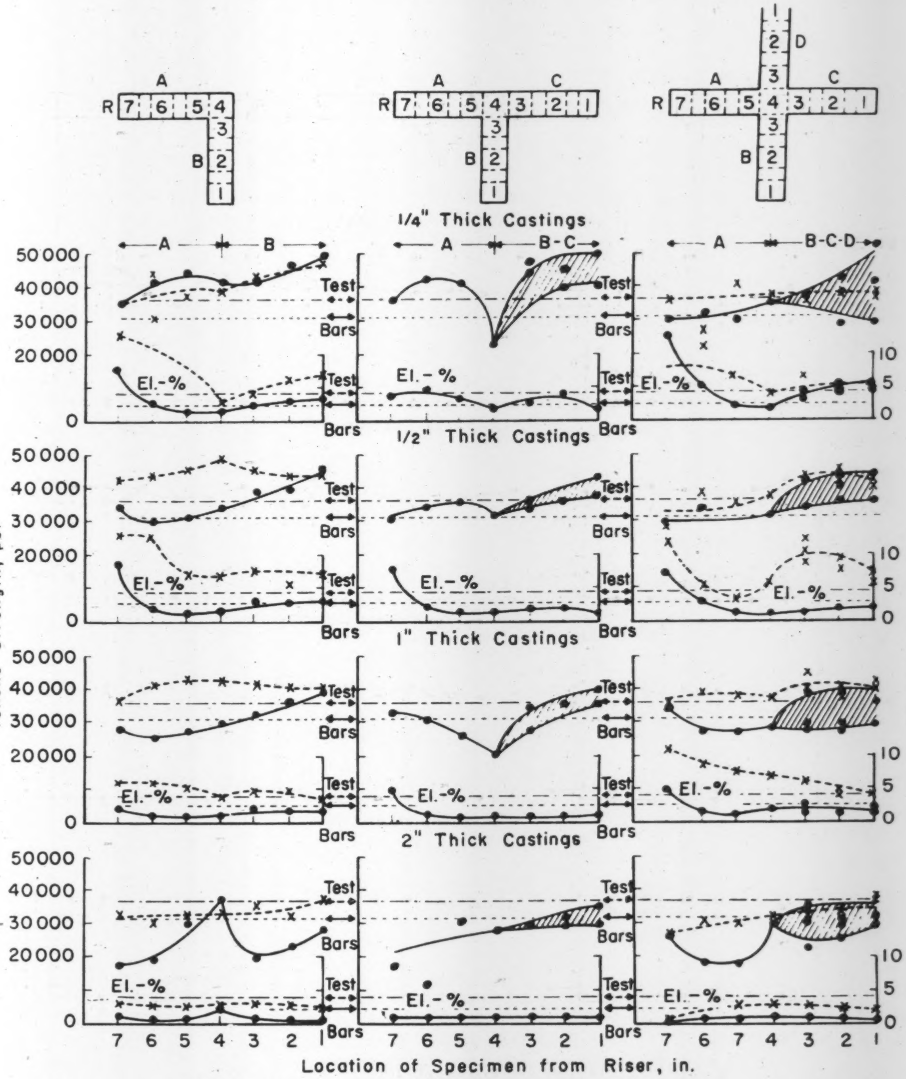

FIG. 7.—Properties in C4A-T6 Joined Section Castings.
Broken curves refer to castings made by means of modified processes.

with the strength properties being relatively uniform throughout any given casting. Marked surface-to-center strength differentials within the SG70A alloy castings occur only in the 12 by 17-in. plates of 1 and 2-in. thicknesses. However, these differentials are minimized when insulated risers, which provide for better feeding, are used in place of the conventional sand risers as shown by the cross-hatched bands of Fig. 5. No significant improvement resulted from the use of the insulated risers on the ½-in. thick plates in this series.

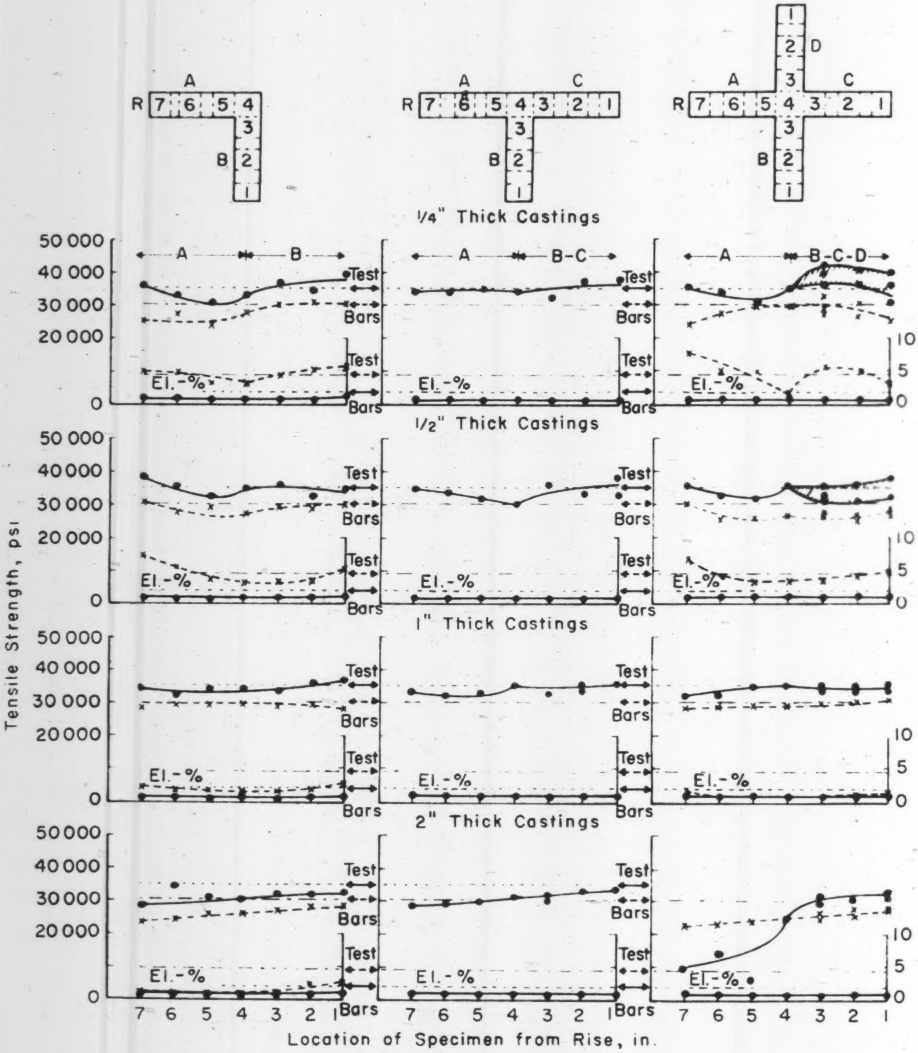

FIG. 8.—Properties in SG70A-T6 Joined Section Castings.

Broken curves refer to castings made by means of modified processes.

The resultant effects of adding grain refiners to the melts, pouring the castings in the vertical position, and changing gating and risering practices was to produce more uniform properties throughout the C4A alloy castings (Fig. 6). In general, the second series of C4A alloy castings and separately-cast test bars which were made according to the techniques described above had higher tensile strengths than the first series; the opposite effect resulted in the SG70A alloy castings. These techniques produced higher elongation values for the case of both alloys.

TABLE III.—RELATION OF CASTING TO SEPARATELY CAST TEST BAR TENSILE STRENGTHS.

Thickness, in.	L, T, X Sections		Plate Sections		All Castings	
	Average Tensile Strength, psi	Percentage of Test Bar Tensile Strength	Average Tensile Strength, psi	Percentage of Test Bar Tensile Strength	Average Tensile Strength, psi	Percentage of Test Bar Tensile Strength
			Alloy C4A			
¼	41 000	129	34 600	109	37 800	119
½	35 200	111	35 600	112	35 100	111
1	32 000	101	32 400	102	32 200	102
2	26 400	83	30 800	97	30 000	90
			Modified Mold and Melt Processing			
¼	38 100	105	37 000	102	37 600	103
½	42 600	117	40 100	110	41 400	104
1	39 600	109	39 000	107	39 300	108
2	32 400	89	32 300	89	32 400	89
			Alloy SG70A			
¼	34 300	98	36 300	104	35 300	101
½	34 500	99	35 800	102	35 200	101
1	34 400	98	33 000	94	33 700	96
2	29 200	83	28 000	80	28 600	82
			Modified Mold and Melt Processing			
¼	28 100	91	28 300	92	28 200	91
½	28 000	91	29 900	95	28 700	93
1	29 700	96	30 600	99	30 100	97
2	26 300	85	25 800	83	26 100	85
			Insulated Riser			
½			34 400	98		
1			34 700	99		
2			32 300	92		

Properties obtained from various locations in the L, T, and X sections made of these two alloys are shown in Figs. 7 and 8. The bands for the T and X sections in these figures represent the range of properties obtained from the casting members which were not connected to the riser. There was no trend in property levels observed in these members due to the position of the member—that is, the properties of member *B* in the X sections, for example, did not show properties which were consistently superior or inferior to those in the *C* and *D* members. As was the case for the plate sections, the SG70A alloy joined-section castings, Fig. 8, are characterized by comparative uniformity of tensile properties except for the case of the thickest X section where strengths at near-riser locations are low. This is believed to be an effect similar to that noted in the largest 2-in. plate casting made of this alloy and would undoubtedly be remedied by using plaster risers.

TABLE IV.—RELATION OF CASTING TO SEPARATELY CAST TEST BAR ELONGATION VALUES.

Thickness, in.	L, T, X Sections		Plate Sections		All Castings	
	Average Elongation, per cent	Percentage of Test Bar Elongation	Average Elongation, per cent	Percentage of Test Bar Elongation	Average Elongation, per cent	Percentage of Test Bar Elongation
			ALLOY C4A			
¼	3½	117	1½	50	2½	83
½	2¾	92	1⅔	56	2¼	75
1	1½	50	1¼	42	1½	50
2	1	33	1¼	42	1	33
			MODIFIED MOLD AND MELT PROCESSING			
¼	7	175	6	150	6½	163
½	8¾	220	6¼	156	7½	188
1	5¼	131	4¾	119	5	125
2	3	75	3	75	3	75
			ALLOY SG70A			
¼	¾	25	¾	25	¾	25
½	¾	25	1½	50	1	33
1	1	33	1	33	1	33
2	1	33	1	33	1	33
			MODIFIED MOLD AND MELT PROCESSING			
¼	5	111	4	89	4½	100
½	4½	100	3½	78	4	89
1	1¾	39	1¾	39	1¾	39
2	1¼	28	1½	33	1½	33
			INSULATED RISER			
½			¾	25		
1			1	33		
2			¾	25		

In the case of the C4A alloy, the T, X, and L castings also exhibit the same trends observed in the simple plate castings, with low strengths existing near the riser and higher strengths existing at casting extremities. It may be noted that the tensile strengths throughout these castings are quite erratic; however, the L and X castings made with the modified molding practices and grain refiners exhibit more uniform properties as indicated by the broken curves. This latter group of castings also appears to be less sensitive to edge and riser effects insofar as tensile properties are concerned.

Summary relationships between the

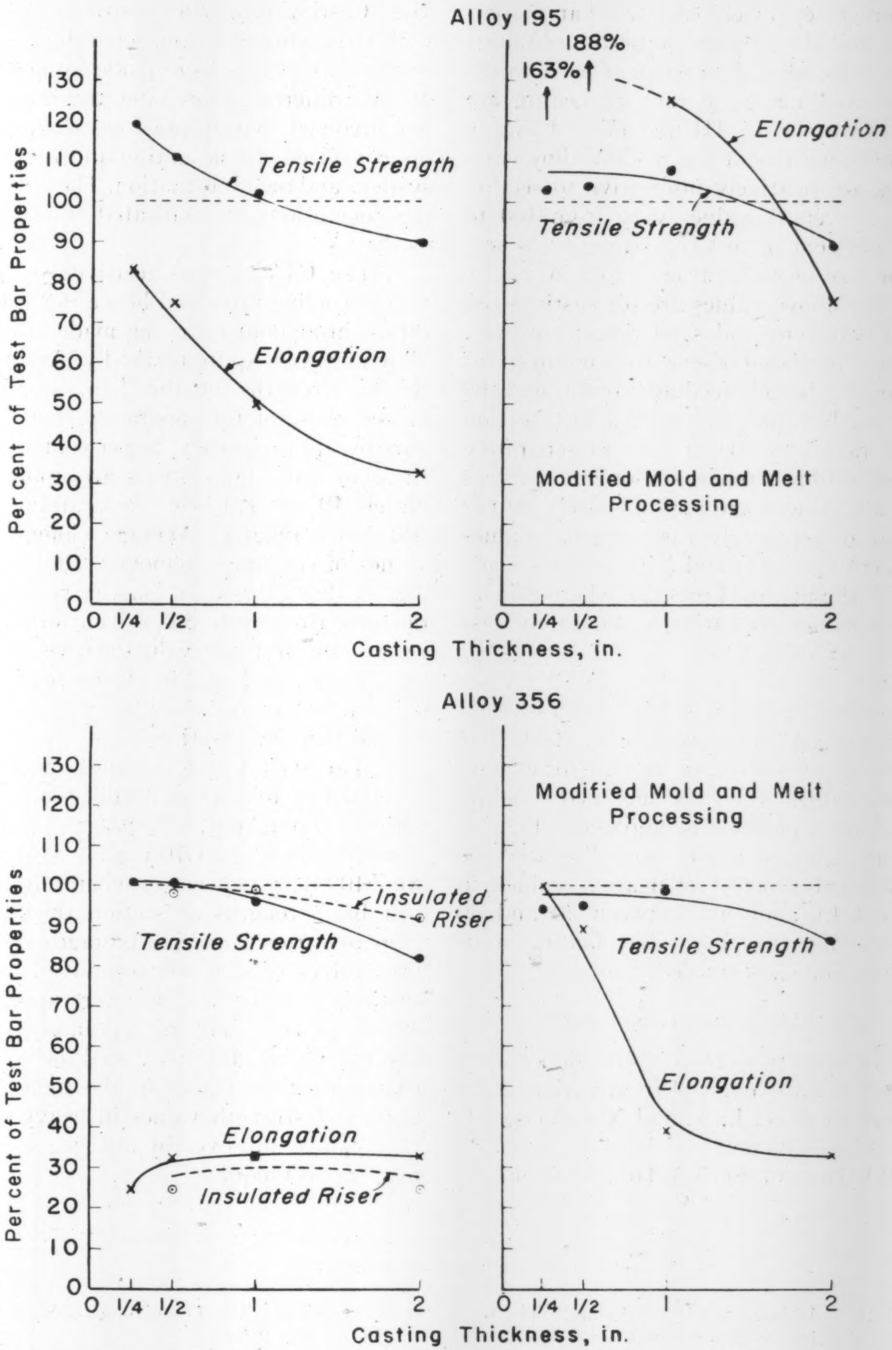

FIG. 9.—Summary Relationships of Average Casting to Average Test Bar Properties.

average separately cast test bar properties and the average properties obtained from the several castings of each thickness and made by each procedure are shown in Tables III and IV and Fig. 9. The properties of the SG70A alloy castings are relatively insensitive to section size. Strength values vary from 100 to 80 per cent of test bar strengths as section size increases from $\frac{1}{4}$ to 2 in.

The above values are for castings fed with conventional sand risers; however, when insulated risers are employed to provide better feeding conditions the strength values in the heaviest section are raised to 90 per cent of separately cast test bar strengths. Elongation values of all sections are approximately 30 per cent of separately cast test bar values except for the $\frac{1}{4}$- and $\frac{1}{2}$-in. sections made with the modified practice where elongation values approximate separately cast test bar values.

Strength values in the C4A alloy castings in thicknesses of 1 in. and less equal or exceed separately cast test bar strengths; this is also the case for elongation values when the modified molding and melt practice is employed (Fig. 9). For castings made according to the standard practice elongation values in the C4A alloy are between 80 and 30 per cent of test bar values for the range of section sizes studied.

SUMMARY AND CONCLUSIONS

Tensile properties were determined from blanks removed from various locations in plate, L, T, and X shaped castings of different thicknesses made of C4A-T6 and SG70A-T6 aluminum alloys. Casting properties were compared with those obtained from separately cast test bars. Castings were made by means of two different processing techniques; one involved routine practices while the other utilized special gating and risering systems and melt inoculation. The following conclusions are indicated from this work:

1. The C4A alloy is relatively sensitive to cooling rates and hence the properties throughout a casting made of this alloy may vary quite markedly. Average tensile strengths in the $\frac{1}{4}$-in. sections exceed those of the separately cast test bars by approximately 20 per cent while those in the 2-in. sections are approximately 10 per cent below separately cast test bar strengths. Average elongation values of specimens removed from castings made with routine foundry practices decrease from 80 to 30 per cent of separately cast test bar values with section size changes of $\frac{1}{4}$ to 2 in. These relationships may be improved by the use of special foundry practices.

2. The SG70A alloy castings are characterized by relative uniformity of properties throughout. Average tensile strengths in the SG70A alloy castings were 100 to 80 per cent of separately cast test bar strengths as section thickness increased from $\frac{1}{4}$ to 2 in. Average elongation values of specimens removed from castings of each thickness were approximately 30 per cent of separately cast test bar values. It is indicated, however, that elongation values in the light sections and strength values in heavy sections can be improved by utilizing special foundry techniques.

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DISCUSSION

CHAIRMAN JOHN R. TOWNSEND[1].—The conclusion is that the 195 alloy is much more sensitive than the 356 alloy to casting conditions. This is borne out in our experience at Sandia. Just how is it proposed to use test bars as a measure of testing quality?

MR. W. H. JOHNSON (*author*).—It is unfortunate that the separately cast test bars are not more indicative of the physical properties within castings. If they were, the answer to this question would be relatively simple. It is well known that at best the properties obtained from such bars are an indication of melt quality only, and, consequently, fail to reveal the true measure of casting quality. Until further investigations on casting performance reveal the extent of correlation existing between casting quality and the data obtained from separately cast test bars, this question cannot be answered adequately. At present we must still rely upon separately cast test bar data supplemented, when feasible, by the data obtained by the sectioning of selected castings.

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NOTCH AND SMOOTH BAR STRESS-RUPTURE CHARACTERISTICS OF SEVERAL HEAT-RESISTANT ALLOYS IN THE TEMPERATURE RANGE BETWEEN 600 AND 1000 F*

BY J. G. SESSLER[1] AND W. F. BROWN, JR.[2]

SYNOPSIS

Smooth and sharply-notched bar stress-rupture tests were carried out on a number of heat-resisting alloys in the temperature range between 600 and 1000 F for rupture times up to 1000 hr. The following alloys were tested: (1) the low-alloy chromium-molybdenum-vanadium steel 17-22A(S); (2) the martensitic stainless steels AISI 410 and AMS 5616; (3) the modified precipitation-hardening chromium stainless steel 17-4PH; and (4) the super alloys A-286 and Inconel X.

The results indicate that below 800 F, 1000-hr rupture strengths well in excess of 100,000 psi are obtained for the modified ferritic steels and for 17-4PH. In the temperature range between 600 and 800 F, these alloys are directly competitive with A-286 alloy but considerably weaker than Inconel X. Regarding notch rupture sensitivity, both Inconel X and A-286 exhibit notch rupture strength ratios less than 1.2 at temperatures between 600 and 800 F. The alloy 17-4PH exhibits severe notch sensitivity in this temperature range when tempered at 1000 F. For the remainder of the alloys, indications of developing notch sensitivity are observed at testing temperatures of 800 F and above.

Evidence is presented that the moderately elevated temperature rupture strength of modified ferritic steels can be considerably increased by heat treating to higher strength levels. However, a practical strength limitation appears to exist in that there is a progressive development of notch rupture sensitivity as the strength level is increased.

Present applications of heat-resistant alloys in the aircraft turbine generally involve temperatures well in excess of 1000 F and service lives of only a few hundred hours. Operating temperatures and stresses are sufficiently high to rule out completely many ferritic and austenitic heat-resistant alloys widely used in the steam power and chemical processing industry.

Recently, however, new developments are contemplated in several fields involving lower metal temperatures than encountered in the present aircraft turbine. For example, for commercial aircraft applications it may be desirable to increase the operating life and reliability of hot components in the gas turbine by a reduction in inlet gas temperature possibly combined with turbine cooling. Another problem of current interest is the design of aircraft subjected to aerodynamic skin heating. In some cases, the metal may be required to withstand sustained temperatures sufficiently high to exclude the use of aluminum or titanium alloys.

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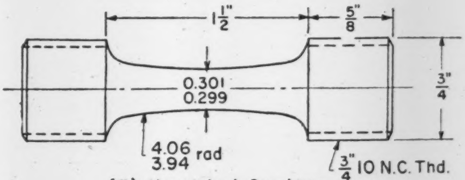
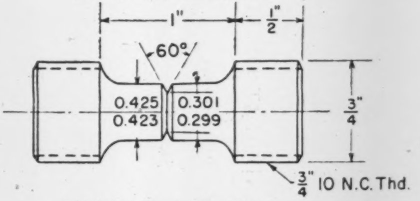
[2] Lewis Flight Propulsion Laboratory, National Advisory Committee for Aeronautics, Cleveland, Ohio.

The metal temperatures of interest in these relatively new fields extend from the lowest temperature at which creep must be considered to temperatures around 1000 F. Thus, the operating temperatures overlap those encountered in the steam turbine industry. However, the environmental conditions are quite different and because of weight considerations the design stresses will be considerably higher. Furthermore, the possibility of exceeding the design temperature during operation (for example, failure of the cooling system) must be kept in mind. At these moderately elevated temperatures, the ferritic heat-resistant steels are in competition with the very costly super alloys, and at sufficiently low temperatures the ferritic materials may offer strength advantages. Of particular interest are the low-alloy and the martensitic stainless steels modified by additions of vanadium, tungsten, or boron coupled with increased carbon contents up to 0.30 per cent. These new compositions possess much higher creep strength and corrosion resistance at moderately elevated temperatures than do the older carbon - molybdenum, chromium - molybdenum, and 12 chromium stainless steels.

An immediate problem facing the designer is the selection of the most suitable alloys for use in a given range of moderately elevated temperatures. Unfortunately, there is very little published information relating to the creep or stress-rupture properties of heat-resistant alloys in the temperature range below 1000 F. In 1950 Smith, Seens, and Dulis (**1**)[3] determined the creep and stress rupture characteristics at 500, 600, and 700 F of SAE 4340 and a nickel-chromium-molybdenum-vanadium steel both hardened to 43 Rockwell C. This work was later extended to 1000 F (**2**). These authors show that 1000-hr rupture strengths in excess of 100,000 psi at 700 F are readily obtainable, and that at lower temperatures the 1000-hr rupture strength exceeds the yield strength. This investigation did not include the newly developed ferritic steels or super alloys. The new ferritic creep-resisting steels possess a considerably greater stability than do the constructional low alloy steels (for example, SAE 4340) and may therefore be heat treated to relatively higher hardness levels for elevated temperature applications. The question arises as to whether certain strength limitations exist that would place an upper limit on the hardness. For example, the hardness of constructional low-alloy steels for use at temperatures where creep does not



(a) Unnotched Specimen



(b) Notched Specimen
Notched rad < 0.001

FIG. 1.—Stress Rupture Specimens.

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 752.

TABLE I.—COMPOSITION AND HEAT TREATMENT OF INVESTIGATED ALLOYS.

Alloy	Composition per cent								Heat Treatment	Rockwell Hardness, C Scale
	C	Cr	Ni	Mo	Mn	Si	Fe	Others		
SAE 4340..........	0.39	0.72	1.82	0.28	0.71	0.29	Bal		1550 F, 1 hr, oil: 1170 F, 2 hr	30
SAE 4340..........	0.40	0.82	1.82	0.24	0.70	0.29	Bal		1550 F, 1 hr, oil: 850 F, 2 hr, air	43
17-22A (S)[a]........	0.30	1.25	0.34	0.51	0.62	0.68	Bal	0.25—V	1725 F, ½ hr, air: 1200 F, 6 hr, air	33
AMS 5616.........	0.12	12.88	1.98	0.50	0.35	0.31	Bal	2.86—W	1800 F, 2 hr, air: 1050 F, 2 hr, air	36
17-4PH[a]...........	0.05	17.86	4.43	...	0.64	0.33	Bal	3.91—Cu 0.36—Cb + Ta	1900 F, 1 hr, air: 1000 F, 1 hr, air	37
AISI 410[b].........	0.15 max	12	...	...	1.00	1.00	Bal		1800 F, ½ hr, air 1050 F, 2 hr, air	...
Inconel X[b].........	0.08	15	72	...	0.70	0.50	7	2.5—Ti 1.0—Cb 0.7—Al	2100 F, 4 hr, oil 1550 F, 24 hr, air 1300 F, 20 hr, air	32
A286[b].............	0.08 max	15	25	1.25	1.50	1.00 max	Bal	2.0—Ti 0.25—V 0.30—Al	1800 F, 1 hr, air blast: 1325 F, 16 hr, air	26

[a] These alloys were also subjected to special heat treatments; see Figs. 10 to 13.

[b] Nominal composition.

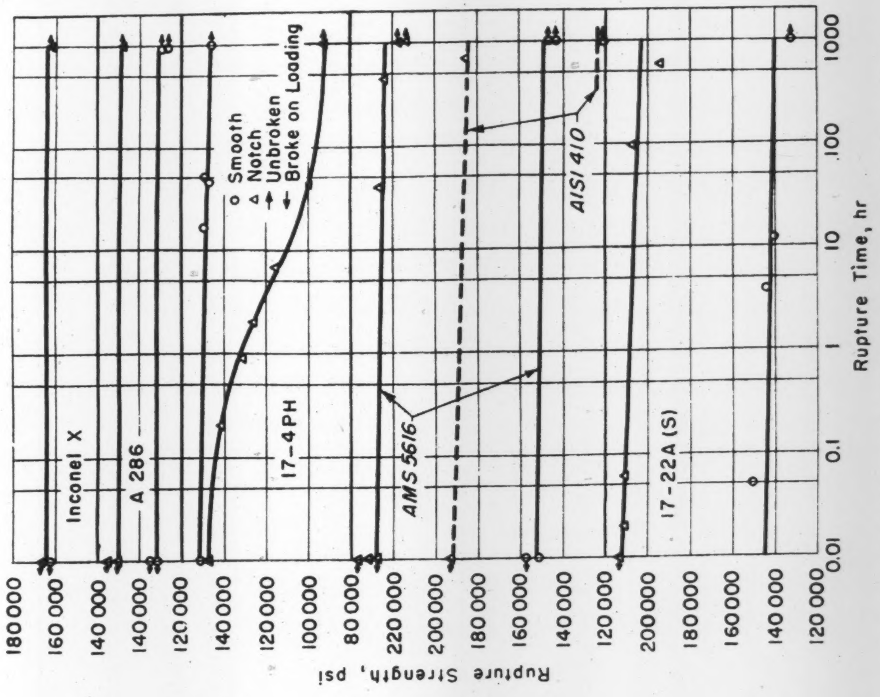

FIG. 2.—600 F Notch and Smooth Rupture Strength for Several Alloys.

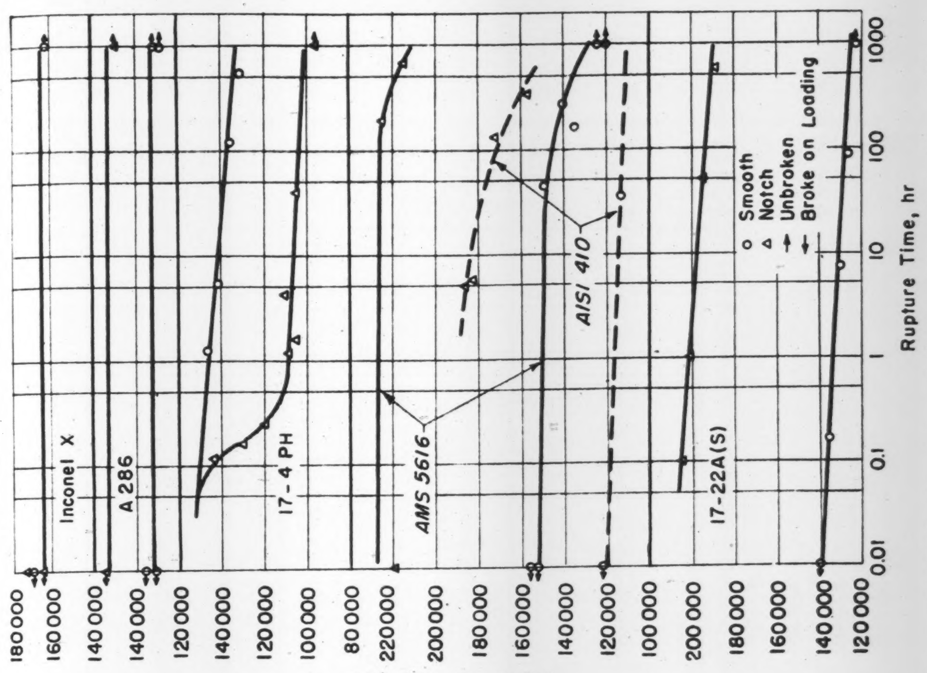

FIG. 3.—700 F Notch and Smooth Rupture Strength for Several Alloys.

occur is limited by a sensitivity to stress concentrations which develops rapidly as the hardness is increased above a certain value. Notch sensitivity has also been observed in the creep test for a large number of alloys tested at temperatures of 1000 F and above (3, 4). However, the dependence of this sensitivity on the initial hardness has received only a limited amount of attention (8).

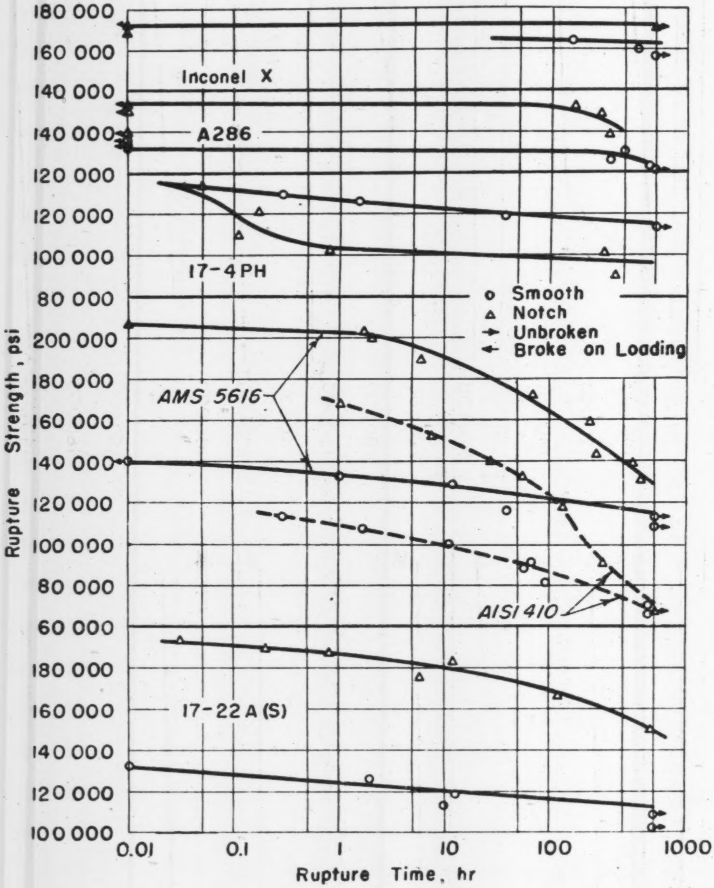

Fig. 4.—800 F Notch and Smooth Rupture Strength for Several Alloys.

The object of the present investigation was to provide the following information: (*1*) stress-rupture data between 0.1 and 1000 hr for several heat-resistant steels and super alloys in the temperature range between 600 and 800 F; (*2*) the influence of sharp notches on the rupture strength of these alloys; (*3*) possible strength limitations at moderately elevated temperatures.

Material and Procedure

The alloys chosen are representative of four classes of creep resistant materials: (*1*) low-alloy steels, represented by 17-22A(S); (*2*) martensitic stainless steels represented by AISI 410 and

AMS 5616; (*3*) modified ferritic chromium stainless steels represented by 17-4PH[4] and; (*4*) super alloys represented by A-286 and Inconel X.

All materials were received in the form of bar stock with diameters between $\frac{3}{4}$ and $1\frac{5}{8}$ in. The chemical composition of these alloys and the heat treatments employed are shown in Table I. These heat treatments conform to the manufacturer's recommended practice. In addition, two of the alloys were subjected to heat-treating procedures designed to provide either higher strength or reduced creep embrittlement.

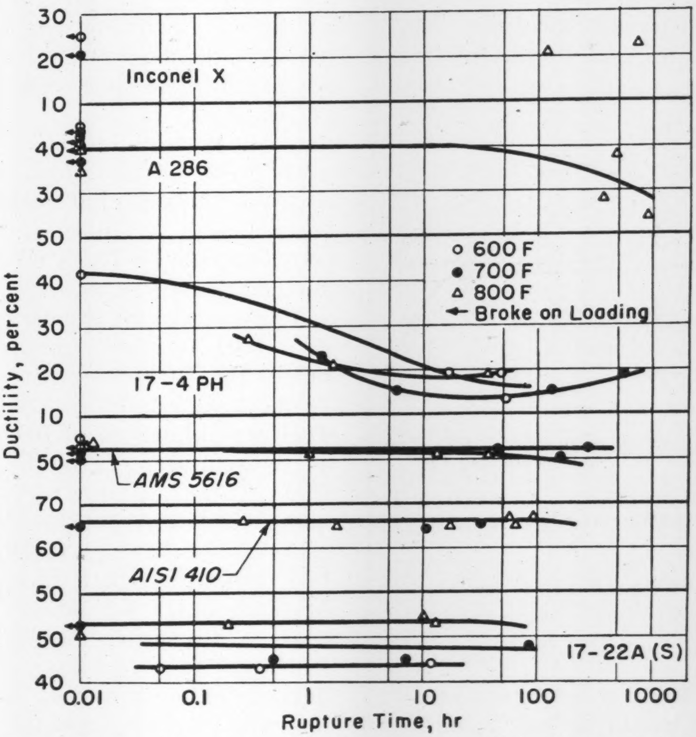

Fig. 5.—Smooth Rupture Ductility as Function of Rupture Time for Several Alloys Tested at 600, 700, and 800 F.

Smooth and sharply notched specimens of the type shown in Fig. 1 were machined from completely heat-treated blanks and tested at 600, 700, and 800 F. The details of specimen preparation and testing technique have been described elsewhere (**3**, **4**).

Results and Discussion

The primary stress-rupture data for notch and smooth specimens tested at 600, 700, and 800 F is plotted as a function of the rupture time in Figs. 2 to 4. Smooth rupture ductility[5] has been similarly represented in Fig. 5. Elevated temperature 0.2 per cent yield strengths are shown in Fig. 6 for all alloys except AISI 410 and 17-4PH. It should be noted that most yield strength data were taken from the literature and therefore cannot be considered to represent exactly the alloy heats tested in this investigation. The SAE 4340 rupture data reported by Smith *et al.* (1, 2) are introduced into the following discussion. While this alloy is not designed specifically for elevated temperature service, the data illustrate some of the difficulties which may be expected when structural steels are used under creep conditions.

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[4] This is a recently developed precipitation hardening alloy. It contains some martensite in its hardened structure and is on the borderline between a ferritic and austenitic material.

[5] Ductility is defined as per cent reduction of area at fracture.

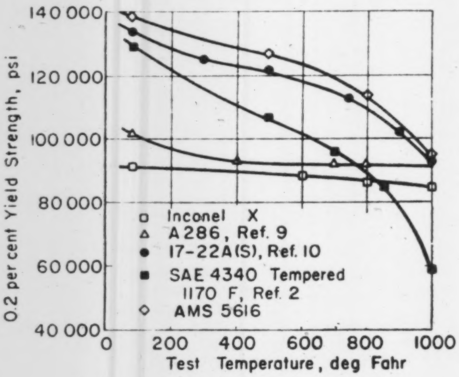

FIG. 6.—Yield Strength as a Function of Test Temperature for Several Alloys.

Smooth Stress Rupture Properties:

At the lowest test temperature, Fig. 2, all alloys except Inconel X and A-286 exhibit the time dependent strength behavior characteristic of creep. The 600 and 500F rupture strength Figs. 2 and 3, of both Inconel X and A-286 is essentially time independent up to 1000 hours.

For the purpose of comparing the alloys, the 1000-hr rupture strength is plotted against the testing temperature in Fig. 7. Previously published results (3, 4, 5) have been used to extend these curves to 1000 F. As might be expected, both Inconel X and A-286 exhibit practically constant rupture strength at temperatures below approximately 800 F. Above this temperature, their strength decreases rather rapidly with increasing temperature. The remaining alloys are characterized by continuously decreasing strength with increasing temperature over the entire range investigated. This rate is practically identical for 17-22A(S), AMS 5616, and 17-4PH. The SAE 4340 and AISI 410 weaken more rapidly with increasing temperature. At temperatures above 800 F the alloys fall in essentially the same order of strength as observed in higher temperature tests (3, 4), with the modified ferritic steels being considerably superior to the structural steels but weaker than the super alloys. At temperatures between 600 and 800 F, Inconel X is considerably stronger than the other alloys, possessing a 1000-hr rupture strength of better than 160,000 psi. In this temperature range the 1000-hr rupture strength of the modified ferritic steels and 17-4PH is well in excess of 100,000 psi and at temperatures of 700 F and below these alloys are directly competitive with A-286. SAE 4340 tempered to a Rockwell C hardness of 43 shows very high rupture strength at temperatures below about 600 F. However, structural steels at this hardness level possess poor stability. The results are interesting, nevertheless, because they indicate that improved rupture strengths at moderately elevated temperatures can probably be obtained by increasing the hardness of the more stable modified ferritic steels.

It is interesting to compare the 1000-hr rupture strengths, Fig. 7, with the elevated temperature yield strengths shown in Fig. 6. In the temperature range between 600 and 1000 F, the yield strengths of the two super alloys A-286 and Inconel X are practically independent of temperature. For Inconel X and A-286 the yield strength is below the 1000-hr rupture strength up to 1000 F and 900 F respectively. The yield strengths of the modified ferritic steels

17-22A(S) and AMS 5616 fall continuously with temperature, but at a slower rate than observed for SAE 4340 tempered at 1170 F. The yield strengths of 17-22A(S) and AMS 5616 are lower than the 1000-hr rupture strength at temperatures below about 800 F.

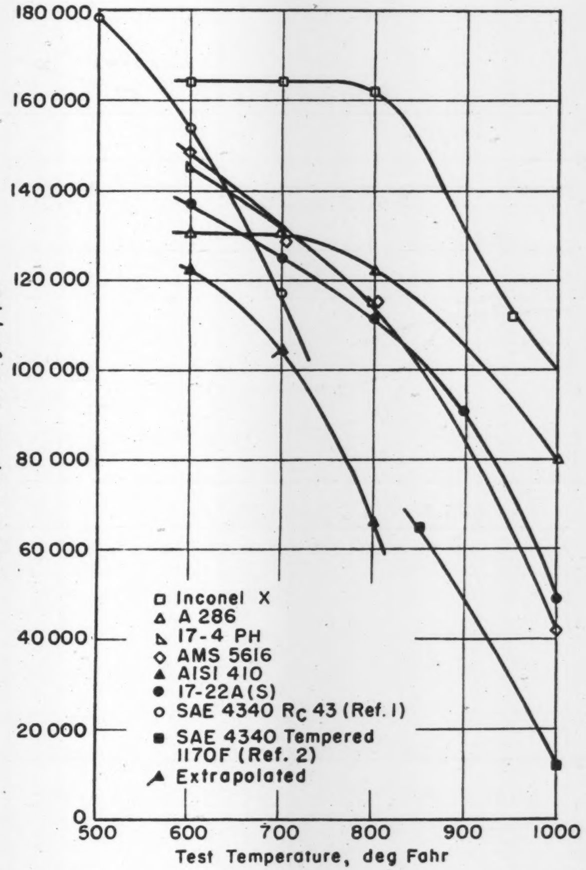

Fig. 7.—1000-hr Rupture Strength as a Function of Test Temperature for Several Alloys.

The rupture ducitility levels, Fig. 5, with the exception of A-286 and 17-4PH, are quite insensitive to either temperature or rupture time in the range between 600 and 800 F. While the data for 17-4PH is rather incomplete, the ductility appears to exhibit a time-temperature dependence similar to that observed for other heat-resistant alloys at higher temperatures. The ductility of A-286 at 800 F appears to be decreasing at times above about 200 hr. In all cases the ductility values are relatively high, the lowest being about 15 per cent exhibited by 17-4PH.

Notch Sensitivity:

The notch strength as a function of rupture time at 600, 700, and 800 F is shown in Figs. 2 to 4. In order better to evaluate notch sensitivity, these data have been re-plotted in Fig. 8 with the notch rupture strength ratio as a func-

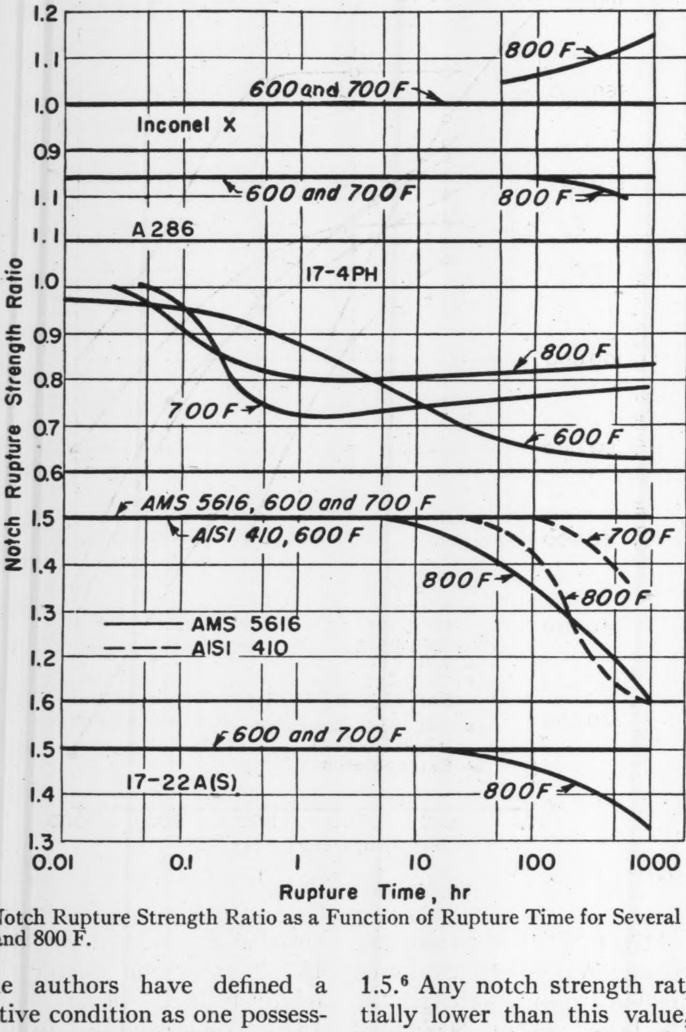

FIG. 8.—Notch Rupture Strength Ratio as a Function of Rupture Time for Several Alloys Tested at 600, 700, and 800 F.

tion of the rupture time. The presence or absence of notch sensitivity has generally been judged from an examination of this ratio. Many investigators including the authors have defined a notch-sensitive condition as one possessing a notch strength ratio below unity. However, such a definition is quite arbitrary and does not consider the known fundamental influence of notches on a metal. It has been shown by Sachs *et al.* (6) that the notch strength ratio of fully notch-ductile structural low-alloy steels at room temperature approaches a constant value of about 1.5[6] at low hardness levels. Similarly, it has been observed (5) that the notch rupture strength ratio of a sufficiently ductile stable alloy is also approximately 1.5.[6] Any notch strength ratio substantially lower than this value, therefore, indicates presence of notch sensitivity. It is realized that many high-strength alloys are most effectively used in a condition yielding a notch strength ratio considerably less than 1.5. However, it is to be emphasized that these applications have been the result of careful development and that in general the safe lower limit of the notch strength ratio will depend on both the material and service condition.

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[6] This value was obtained using 50 per cent sharp notches (see Fig. 1). It will vary with notch geometry, and to a lesser extent with the alloy.

The results for the super alloys Inconel X and A-286 are interesting in that notch strength ratios appreciably less than 1.5 are observed even at the lowest testing temperatures where creep is negligible. The notch rupture strength ratio for Inconel X is shown in Fig. 9 as a function of testing temperature with rupture time as parameter. These results, covering a very wide range of temperatures, are similar to those previously observed for 17-22A(S) (5) in that the notch strength ratio curves exhibit minima which occur at progressively lower temperatures as the rupture time increases. In contrast to the behavior of 17-22A(S) the notch strength ratio curves do not continuously rise from their minimum value to approximately 1.5 at low testing temperatures. Instead the notch strength ratio of Inconel X increases to a maximum value and then decreases to approximately unity at temperatures below 700 F. Apparently for both Inconel X and A-286 a fully notch-ductile condition is not obtained at moderately elevated temperatures.

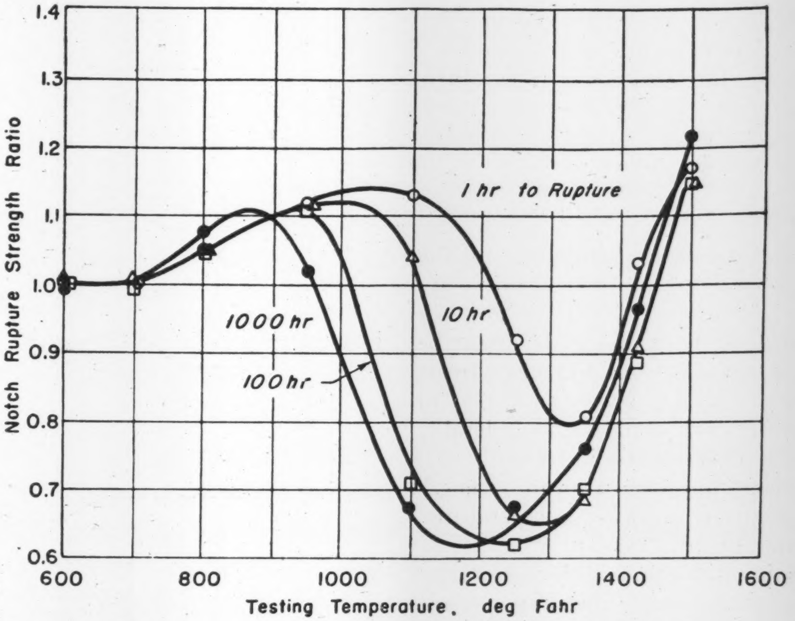

FIG. 9.—Notch Rupture Strength Ratio for Inconel X as a Function of Test Temperature with Rupture Time as Parameter.

The ferritic alloys 17-22A(S) and AMS 5616 are known to develop a time-temperature-dependent notch sensitivity at temperatures over 1000 F (4, 5). According to Fig. 8, these alloys exhibited a notch rupture strength ratio of about 1.5 up to 1000 hr at 600 and 700 F. At 800 F, the notch rupture strength ratio falls with increasing rupture time, reaching a value of approximately 1.3 for 17-22A(S) and 1.1 for AMS 5616 at 1000 hr. The data for AISI 410 indicate that a time-temperature-dependent notch sensitivity begins to develop at temperatures as low as 700 F and results in a 1000-hr notch rupture strength ratio of 1.1 at 800 F.

The results for 17-4PH, are very interesting in that a large notch sensitivity develops in very short rupture times at temperatures approaching the lower limit of the creep range. This notch sensitivity is similar to that previously observed for many other high-temperature alloys in that it appears to be time-temperature dependent. However, for all other alloys studied by the authors, strong notch sensitivity is always observed in a temperature range where the creep behavior is well developed.

POSSIBLE STRENGTH LIMITATIONS

The heat treatments employed for the alloys studied in this investigation are designed primarily for service applications at 1000 F or above. It might be expected that by suitable heat treatment the strength level (hardness) of the ferritic steels could be considerably increased. This increase in strength level should yield improved rupture strength at moderately elevated temperatures. The question arises as to whether increases in strength will be associated with a decrease in ductility or appearance of notch sensitivity. Thus, there may be a practical limitation to the strength level which could be employed. Such strength limitations exist for the structural steels used at room temperature. For these alloys, strength levels in excess of approximately 220,000 psi are associated with severe notch sensitivity in spite of high smooth bar ductility (6, 7).

Higher strength levels in ferritic steels may be obtained by: (*1*) an increase in the austenitizing temperature which will increase the degree of solution of the hardening elements, and (*2*) a decrease in tempering temperature. Consider for example the chromium-molybdenum-vanadium steel 17-22A(S). The conventional heat treatment yields a structure which maintains its hardness for at least 1000 hr at 1000 F. With this excellent stability, it should be possible to employ considerably higher strength levels for use at moderately elevated temperatures.

According to previous studies (8), full carbide solution without excessive grain growth can be obtained by normalizing 17-22A(S) at 1850 F for $\frac{1}{4}$ hr. The room-temperature hardness and tensile properties are shown in Fig. 10 as a function of the 6-hr tempering temperature for bar stock normalized $\frac{1}{4}$ hr at 1850 F. It will be noted that the hardness of the alloy remains essentially unchanged up to 1100 F except for a slight secondary hardening in the vicinity of 1000 F. At 1200 F (the conventional tempering temperature) the hardness has dropped. The yield and the

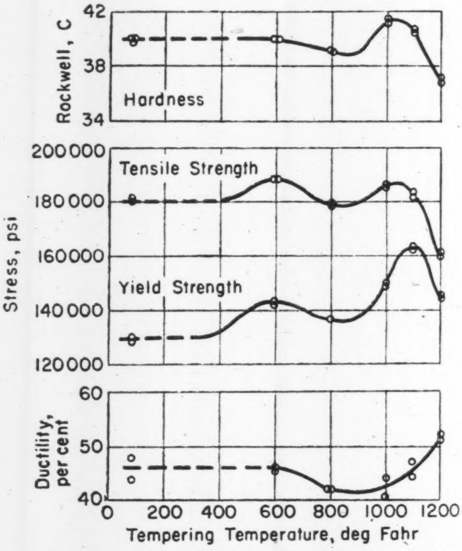

FIG. 10.—Effect of Tempering for 6 hr at Various Temperatures on Room Temperature Properties of 17-22 A(S) Normalized $\frac{1}{4}$ hr at 1850 F.

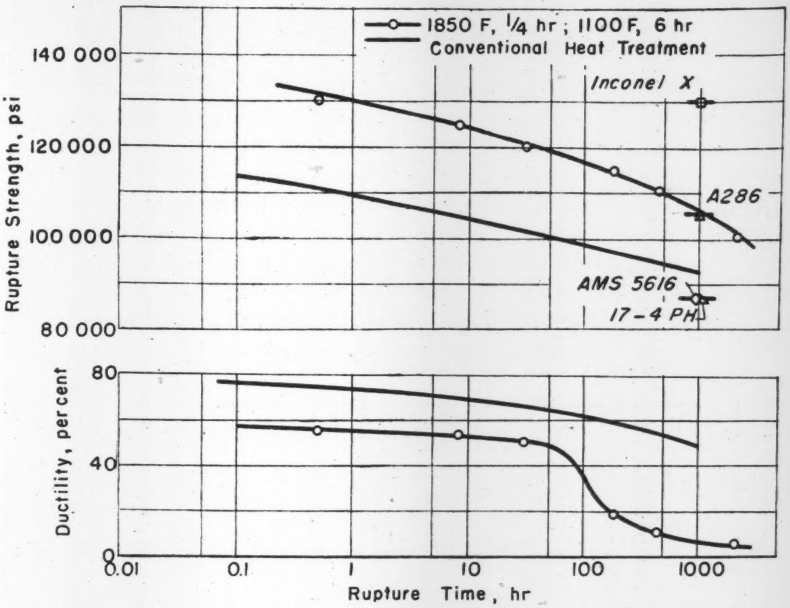

FIG. 11.—900 F Smooth Rupture Strength and Ductility of 17-22 A(S) in Two Conditions of Heat Treatment.

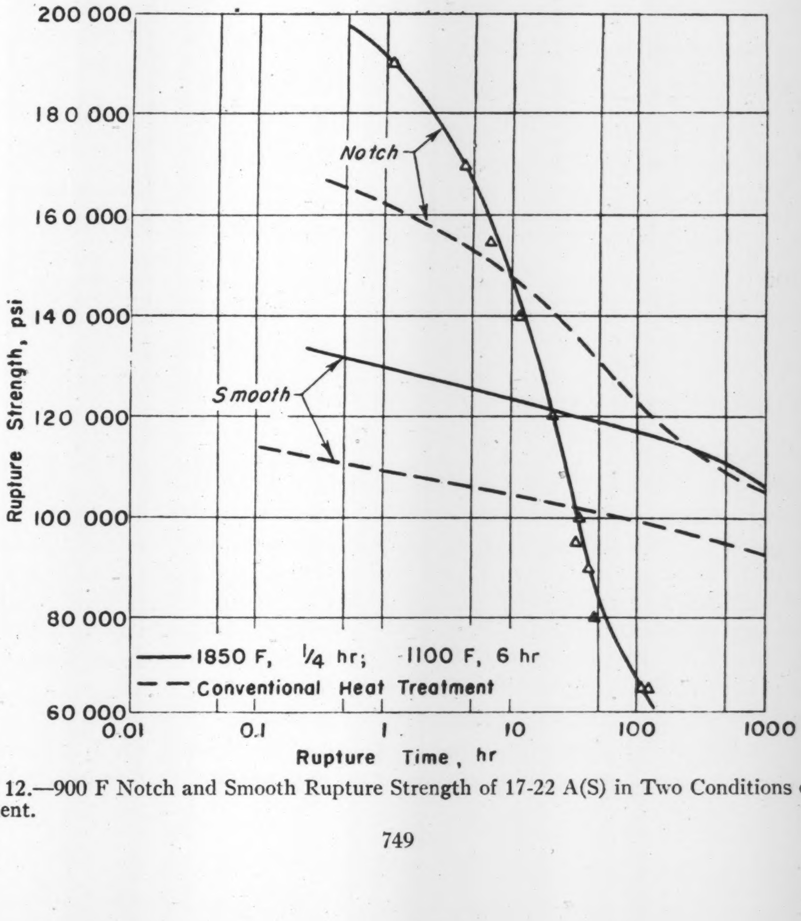

FIG. 12.—900 F Notch and Smooth Rupture Strength of 17-22 A(S) in Two Conditions of Heat Treatment.

tensile strength curves reflect a combination of the relief of residual stress, strain age hardening, and secondary hardening. The important observation is that tempering temperatures in excess of 1100 F produce rapid loss in strength. The ductility is always high but exhibits a shallow minimum in the region of secondary hardening. For moderately elevated temperature service, it would seem reasonable to select a tempering temperature of 1100 F. This yields a Rockwell hardness of about C41 compared with C33 produced by the conventional heat treatment. A sharply notched specimen tempered at 1100 F has a notch strength ratio of 1.45 when when tested at room temperature.

Material given the above described heat treatment was tested in stress rupture at 900 F. The smooth rupture properties are shown in Fig. 11 in comparison with those of the conventionally heat-treated alloy. Added to this representation is the 1000-hr rupture strength of several more highly alloyed materials. The new heat treatment has increased the 1000-hr rupture strength by about 15 per cent, and it is now equal to that of A-286. The conventional heat treatment results in a 900 F rupture ductility which decreases gradually with increasing rupture time to a value of about 50 per cent at 1000-hr. In contrast, the new heat treatment results in a rather rapidly decreasing ductility at rupture times over about 50 hr and a 1000-hr ductility of approximately 10 per cent. However, this alloy would not be considered brittle by conventional standards.

The results of notched 900 F stress-rupture tests on 17-22A(S) given the conventional and the new heat treatment

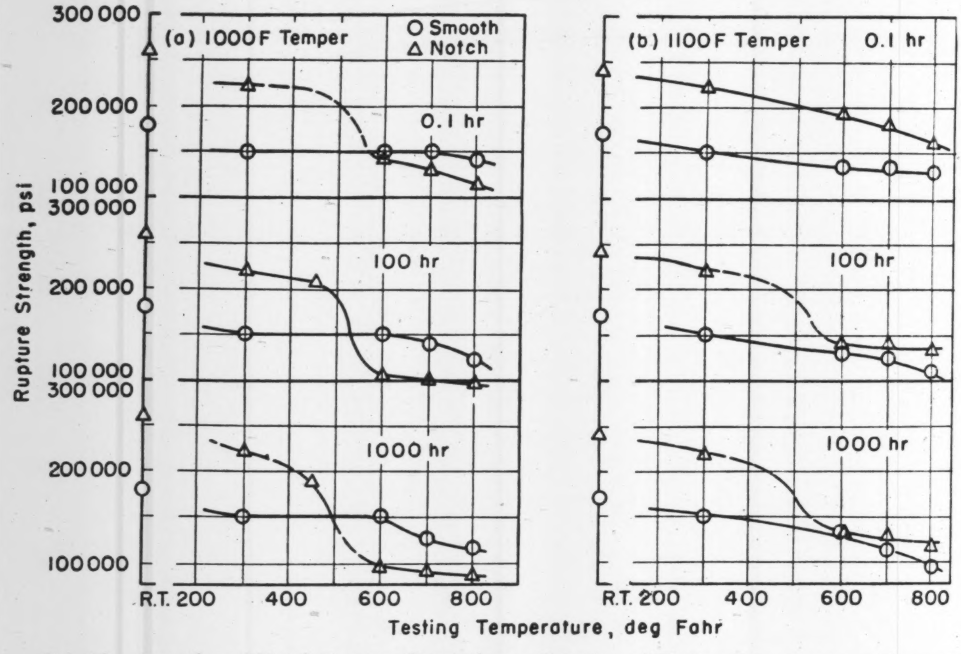

FIG. 13.—Smooth and Notch Rupture Strength as a Function of Testing Temperature at Several Rupture Times for 17-4 PH Tempered at 1000 F and 1100 F.

are compared in Fig. 12. This representation graphically illustrates a strength limitation associated with raising the rupture strength by this heat treatment. The conventional heat-treated material is notch strengthened up to 1000 hr. In contrast, the new heat treatment results in severe notch weakening, the notch strength ratio being 1.45 at 1 hr and only 0.6 at 100 hr. It would have been difficult to anticipate this degree of notch sensitivity from the smooth bar ductility, which is at least 25 per cent at 100 hr to rupture.

On the basis of these results, it may be expected that notch sensitivity in ferritic steels is increased in magnitude and shifted to lower temperatures by employing heat treatments that raise the strength. It might be expected that similar effects would be noted if the content of hardening elements were increased.

The high sensitivity of the notch rupture strength to variations in heat treatment is further illustrated by considering the alloy 17-4PH. The notch and smooth rupture strength of this alloy in two conditions of temper is compared in Fig. 13. It will be noted that the severe notch sensitivity associated with the 1000 F temper is greatly reduced by raising the tempering temperature to 1100 F. This improvement is associated with a relatively small loss in the smooth bar strength.

The previous discussion has perhaps overemphasized the danger of developing notch sensitive structures by attempts to raise the strength through heat treatment. Actually the authors feel that the full strength potential of ferritic steels at moderately elevated temperatures is not realized by the conventional heat treatments. However, the results do emphasize the need for systematic investigations designed to develop optimum combinations of smooth and notch bar properties. The feasibility of optimizing the heat treatment of 17-22A(S) for high-temperature service has been demonstrated previously (8).

CONCLUSIONS

The alloys reported in this paper are intended to represent certain classes that might be of interest for moderately elevated temperature applications. On the basis of these results, it would seem possible to formulate certain conclusions regarding the selection of materials for use in the temperature range between 600 and 1000 F. It is not suggested that alloy selection be based on rupture data alone, and it is realized that a complete picture must include creep and fatigue data.

1. Structural steels such as SAE 4340 may be heat treated to high strength levels. These structures will possess very high rupture strength below 600 F. However, at higher test temperatures the rupture strength decreases rapidly due to lack of stability.

2. The super alloy Inconel X exhibited the highest rupture strength and greatest stability of strength at temperatures between 600 and 1000 F. Alloy A-286 showed similar stability but considerably lower strength in this temperature range.

3. At temperatures below approximately 800 F, the 1000-hr rupture strength of the modified ferritic steels and 17-4PH is well in excess of 100,000 psi, and these materials are definitely competitive in this temperature range with the super alloy A-286.

4. For both the super alloys and the modified ferritic steels, the yield strength is below the 1000-hr rupture strength at temperatures less than approximately 800 F.

5. Both Inconel X and A-286 exhibit notch rupture strength ratios less than 1.2 at temperatures between 600 and 800 F. The alloy 17-4PH tempered at 1000 F shows a very severe notch sen-

sitivity in this temperature range. The remainder of the alloys exhibit indications of developing notch sensitivity at temperatures of 800 F and above.

It appears that the moderately elevated temperature rupture strength of normalized and tempered modified ferritic steels can be considerably improved by increasing their hardness. However, a possible strength limitation associated with this hardness increase is development of notch rupture sensitivity.

Adknowledgement:

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(5) D. P. Newman, M. H. Jones, and W. F. Brown Jr., "Time-Temperature Dependence of the Notch Effect and Influence of Notch Depth in Stress Rupture Tests on a Cr-Mo-V Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 53, p. 677 (1953).

(6) G. Sachs, L. J. Ebert, and W. F. Brown Jr., "Comparison of Various Structural Low Alloy Steels by Means of the Static Notch Bar Tensile Test," T. P. 2110, *Metals Technology*, Vol. 13, no. 8, Dec. 1946.

(7) G. Sachs, G. S. Sangdahl, and W. F. Brown Jr., "New Notes on High Strength Heat-Treated Steels," *Iron Age*, Vol. 166, Nov. 23, p. 59; Nov. 30, p. 76.

(8) M. H. Jones, D. P. Newman, G. Sachs, and W. F. Brown Jr., "Effects of Variation in Normalizing and Tempering Procedure on Stress Rupture Strength, Creep Embrittlement and Notch Sensitivity for a Cr-Mo-V and a 17 Cr-4Ni-4Cu Steel," *Transactions*, Am. Soc. Metals, Vol. 47, p. 926 (1955).

(9) "Stress Rupture Properties of S-816, S590, and A286 Alloys," Report prepared for ASTM-ASME Joint Committee on the Effect of Temperature on the Properties of Metals, Research Laboratories, Allegheny Ludlum Steel Corp., Watervliet, N. Y., June 1, 1955.

(10) Timken Technical Bulletin, No. 36, 1949, Timken Roller Bearing Co., Canton, Ohio.

DISCUSSION

Mr. W. L. Badger.[1]—It is suggested that if further work is to be done by the authors, that a molybdenum-vanadium modification of type 403—called Lapelloy, or Crucible Steel Co.'s 422 alloy be tested. These steels are more commonly used in the upper part of the temperature range under consideration.

Mr. T. E. Kihlgren.[2]—We should like to call attention to the fact that there are properties other than rupture that would be governing factors in design in the temperature range discussed in this paper, as far as Inconel "X" is concerned. For example, the short-time 0.2 per cent yield strength of Inconel X at 1000 F, using the full heat treatment employed by the authors, is about 84,000 psi, a value much lower than the 1000-hr rupture life of about 110,000 psi. It is apparent that at this temperature the short-time tensile properties would have to be considered for design purposes.

We do not have notch rupture test data at the lower temperatures covered, partly for the above reason and partly because the field of application of this alloy is generally in a higher temperature range. It is certainly true that at higher temperatures of the order of 1350 F, heat treatment can influence considerably the notch rupture strength of high-temperature nickel-base alloys hardened with aluminum and titanium. Increases in notch rupture strength so developed will often be obtained at the expense of some other property.

The authors have stated that the heat treatment used for Inconel X in their tests is that normally recommended for maximum rupture properties above about 1100 F. For applications in the temperature range covered by the authors, this treatment would not normally be applied to Inconel X. The 1625 F stress equalize plus 1300 F aging treatment is employed for applications where service temperatures are below about 1100 F because it results in much better strength and ductility. With the latter heat treatment, the 1000-hr rupture strength usually will again be appreciably higher than the short-time tensile yield strength. It is probable that notch tension test data would be more pertinent than notch rupture data for the temperature range of 600 to 1000 F covered by the authors, as far as Inconel X is concerned.

Mr. Joseph Maltz.[3]—The importance of the moderately elevated temperature range, 600 to 1000 F, to the aircraft industry certainly should not be underestimated. In the airframe field, as contrasted to the engine field, it is very rapidly becoming a serious problem area.

Aerodynamic heating effects have been well publicized in recent years. However, the engine heating effects, particularly in the aft fuselage and in the tail areas, represent the more imminent problem. Aerodynamic heating will not subject structures to the 600 to 1000 F temperature range unless speeds in the order of Mach 3, or approximately 2000 mph, are attained.

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[1] Manager, Thomson Laboratory, Small Aircraft Engine Dept., General Electric Co., W. Lynn, Mass.

[2] Metallurgist, International Nickel Co., Inc., New York, N. Y.

[3] Metallurgist, Bureau of Aeronautics, Navy Dept., Washington, D. C.

The airframe industry is devoting increasingly more attention to the moderately elevated temperature range. The petroleum and steam power plant industries have certainly done a tremendous amount of work on elevated temperature properties, but for the most part they have considered that the properties of the materials they use do not change very much until more elevated temperatures are reached. For aircraft that may not quite be a valid assumption, because of the need to design for minimum weight.

MR. LEO SCHAPIRO.[4]—The authors in their introduction said that the work should have some usefulness to missiles and to transports. If it is going to have any usefulness to missiles or the program that is represented by this work, I think it ought to represent shorter time properties than were presented.

My question is: Does the program involve determining the properties of any shorter times that are of interest to missile people?

MESSRS. J. G. SESSLER AND W. F. BROWN, JR. (*authors' closure*).—The authors wish to thank Messrs. Badger, Kihlgren, Maltz, and Schapiro for their remarks.

Mr. Badger has suggested that Lapelloy and Crucible 422 steel be investigated. Any extension of the program should certainly consider these alloys. Regarding Crucible 422 we have reported previously on its notch stress rupture properties at 1000, 1100 and 1200 F.[5] This alloy is basically similar to AMS 5616. However, it has nearly double the amount of carbon, somewhat more nickel and molybdenum and also contains vanadium. As shown previously,[5] Crucible 422 possesses higher rupture strength than AMS 5616 at temperatures from 1000 to 1200 F even though it was heat treated to a lower initial hardness. Furthermore, the notch rupture sensitivity in this temperature range was distinctly less than that observed for AMS 5616.

Mr. Kihlgren correctly states that Inconel X would possess higher strength at moderately elevated temperatures if given a different heat treatment. The fully heat treated condition was employed in the present investigation so that the data obtained could be appended to that previously reported[6] at temperatures above 1200 F. It is then possible to establish the notch rupture behavior of the same structure over a very wide range of temperature as shown in Fig. 13.

Mr. Kihlgren feels that at temperatures below 1000 F, the yield strength of Inconel X is a more meaningful design parameter than the rupture strength. The authors pointed out in their conclusions that at temperatures below about 800 F the 0.2 per cent yield strength for all the super alloys and modified ferritic steels was below the 1000 hr rupture strength. They do not, however, agree that just because of this fact rupture data are not needed. Design on the basis of yield alone often does not take full advantage of the alloy strength. For example, if the part is subject in service to a rather steep temperature gradient, the over-all deformations may be negligible but the local strain in the hot region quite high. When such is the case, we can use the creep rupture strength or the tensile strength to advantage.

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[4] Chief Metallurgist, Douglas Aircraft Co., Inc., Materials Laboratory, Santa Monica, Calif.

[5] W. F. Brown, Jr., M. H. Jones and D. P. Newman, "Influence of Sharp Notches on the Stress Rupture Characteristics of Heat-Resisting Alloys: Part II," *Proceedings*, Am. Soc. Testing Mats., Vol. 53, p. 661 (1953).

[6] W. F. Brown, Jr., M. H. Jones and D. P. Newman, "Influence of Sharp Notches on the Stress Rupture Characteristics of Several Heat Resisting Alloys," Symposium on Strength and Ductility of Metals at Elevated Temperatures, p. 25, Am. Soc. Testing Mats. (1952). (Issued as special technical publication *ASTM STP No. 128*.)

Mr. Maltz has further emphasized the need to more fully utilize the strength potential of aircraft materials as well as the increasing need for data in the moderately elevated temperature range. We, of course, agree wholeheartedly with him.

Mr. Schapiro feels the data is of no help to the missile people because the rupture times are not sufficiently short. It should be noted that the 0.01-hr (about 30 sec) rupture strengths appear well established. We do not anticipate extension of the program to still shorter times. Certain aspects of missile design do require very short-time data obtained for high rates of loading and heating. There are several laboratories now engaged in gathering this type of data. The usefulness to the missile designer of data reported in this investigation depends on the type of missile and the particular component considered.

INFLUENCE OF HOT-WORKING CONDITIONS ON THE HIGH-TEMPERATURE PROPERTIES OF HEAT-RESISTANT ALLOYS†*

By J. F. Ewing[1] and J. W. Freeman[2]

Synopsis

The relationships between conditions of hot working and properties at high temperatures were investigated for a solution-strengthened 20 chromium, 20 nickel, 20 cobalt, 3 molybdenum, 2 tungsten, 1 columbium alloy. Medium to low strengths will result when large reductions are made at nearly constant high temperatures. Very high strengths at 1200 F, and relatively high strengths at 1500 F are characteristic of gradual reductions over a decreasing temperature range. Strengths equal to those associated with hot-cold working at 1200 F can be obtained by such procedures with finishing temperatures as high as 1800 F. Repeated working with abnormally low reheat temperatures is another cause of very low strengths. Extensive variations in conditions of hot working did not alter the response to subsequent commonly used heat treatments. The extreme sensitivity of lattice parameter to working conditions and cooling rate suggests that unidentified reactions such as ordering of the matrix structure have more effect on properties than now seems evident.

Controlled hot working should be considered seriously as a method for obtaining superior combinations of strength and ductility at elevated temperatures.

Properties of metals and alloys in the hot-worked condition generally are considered difficult to control. These difficulties stem principally from the lack of quantitative information relating conditions of hot-working to properties together with a lack of understanding of the basic principles involved. This paper presents the results of a study covering these features as related to properties at high-temperature for one heat resistant alloy.

It is known that high-temperature strengths ranging from very high to very low values are exhibited by alloys in the hot-worked condition. These extreme variations in properties suggest that control of hot-working conditions could be used as a practical production tool to obtain superior high-temperature strength, just as heat treatment is used today to obtain desired properties.

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† Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

* This paper is based on a thesis by J. F. Ewing submitted in partial fulfillment of the requirements for the degree of Doctor of Philosophy to the University of Michigan.

[1] Research Metallurgist, Babcock & Wilcox Co., Tubular Products Division, Beaver Falls, Pa.

[2] Professor of Metallurgical Engineering and Research Engineer, University of Michigan, Ann Arbor, Mich.

The principles governing the influence of hot-working conditions on the high-temperature strength properties of one type of complex heat-resistant alloy in the hot-worked condition have been studied, as well as the influence of hot-working on response to heat treatment. The study applies primarily to those austenitic alloys dependent upon solution annealing or hot-cold working for superior strength properties at elevated temperature, and not upon strong age-hardening reactions. The particular alloy used was nominally 0.15 carbon, 1.5 manganese, 0.50 silicon, 20 chromium, 20 nickel, 20 cobalt, 3 molybdenum, 2 tungsten, 1 columbium, balance iron.

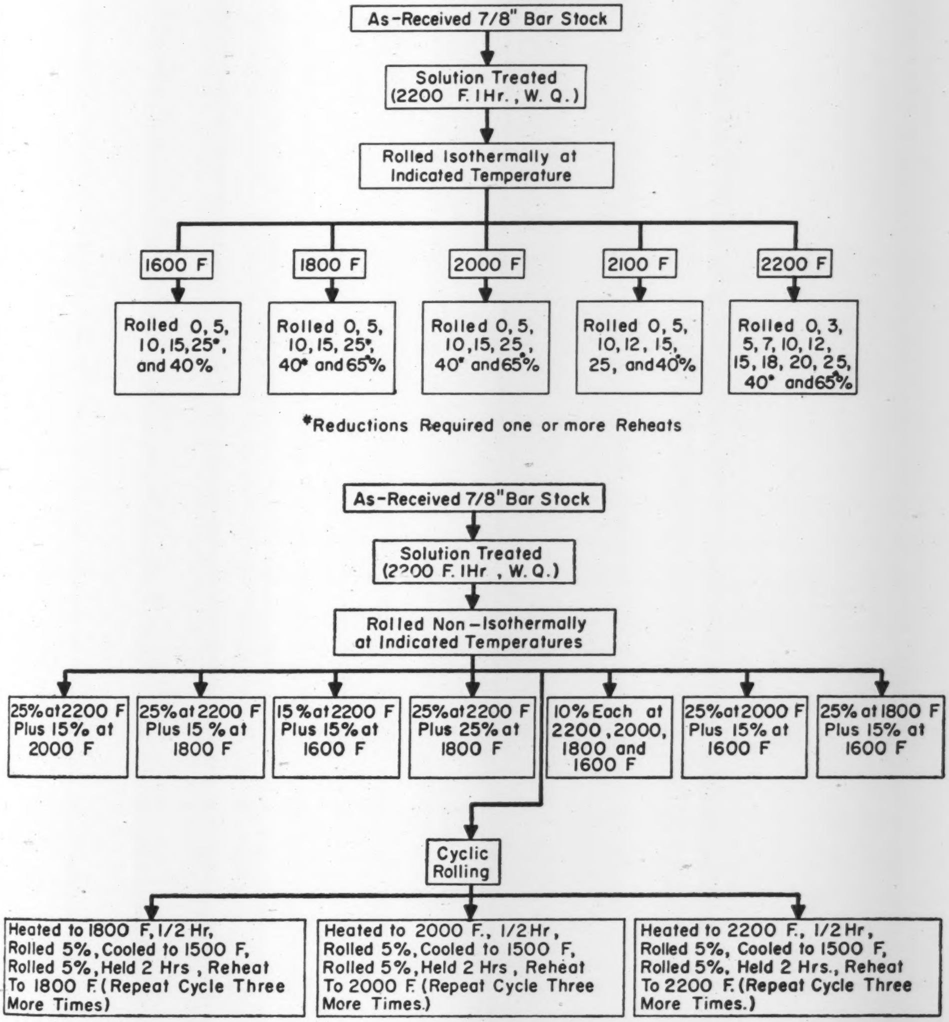

As-Received 7/8" Bar Stock
Solution Treated (2200 F. 1 Hr., W. Q.)
Rolled Isothermally at Indicated Temperature
1600 F
1800 F
2000 F
2100 F
2200 F
Rolled 0, 5, 10, 15, 25*, and 40%
Rolled 0, 5, 10, 15, 25*, 40* and 65%
Rolled 0, 5, 10, 15, 25, 40* and 65%
Rolled 0, 5, 10, 12, 15, 25, and 40%
Rolled 0, 3, 5, 7, 10, 12, 15, 18, 20, 25, 40* and 65%
*Reductions Required one or more Reheats
As-Received 7/8" Bar Stock
Solution Treated (2200 F. 1 Hr, W. Q.)
Rolled Non-Isothermally at Indicated Temperatures
25% at 2200 F Plus 15% at 2000 F
25% at 2200 F Plus 15% at 1800 F
15% at 2200 F Plus 15% at 1600 F
25% at 2200 F Plus 25% at 1800 F
10% Each at 2200, 2000, 1800 and 1600 F
25% at 2000 F Plus 15% at 1600 F
25% at 1800 F Plus 15% at 1600 F
Cyclic Rolling
Heated to 1800 F, 1/2 Hr, Rolled 5%, Cooled to 1500 F, Rolled 5%, Held 2 Hrs, Reheat To 1800 F (Repeat Cycle Three More Times)
Heated to 2000 F., 1/2 Hr, Rolled 5%, Cooled to 1500 F, Rolled 5%, Held 2 Hrs, Reheat To 2000 F (Repeat Cycle Three More Times.)
Heated to 2200 F., 1/2 Hr, Rolled 5%, Cooled to 1500 F, Rolled 5%, Held 2 Hrs., Reheat To 2200 F (Repeat Cycle Three More Times.)

FIG. 1.—Flow Sheet of the Rolling Program.

EXPERIMENTAL PROCEDURE

Rolling of $\frac{7}{8}$-in. square bar stock was used in order to obtain accurate control of working temperatures and amounts of deformation, the two hot-working variables studied. The research program was organized as follows: (1) bars were rolled isothermally varying amounts both above and below minimum recrystallization temperatures to define influences of reduction and temperature on high-temperature strength; (2) stock was rolled non-isothermally over controlled temperature ranges to study influences of decreasing temperatures during hot-working; (3) bars were rolled in cycles between high and low temperatures to measure the influence of extensive precipitation during working on high-temperature strength; (4) material worked under selected conditions were heat treated to study the influence of prior hot working on response to heat treatment. Rupture and creep tests, hardness measurements, metallographic studies, and lattice parameter measurements were used to evaluate the effects and to study the mechanism by which hot working affects high-temperature strength properties. Figure 1 summarizes the hot-rolling program.

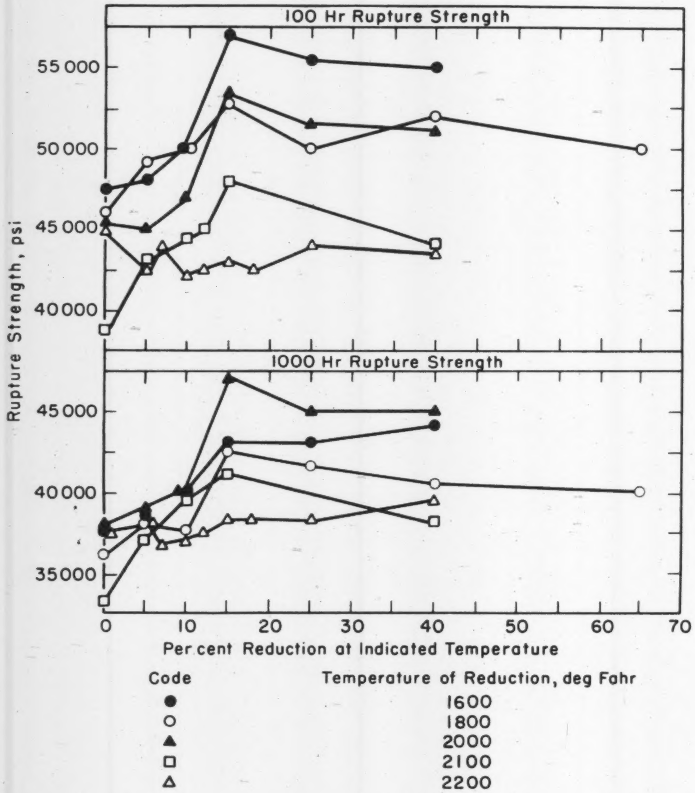


FIG. 2.—Effect of Amount of Isothermal Reduction at Various Temperatures on the 100 and 1000-hr Rupture Strengths at 1200 F.

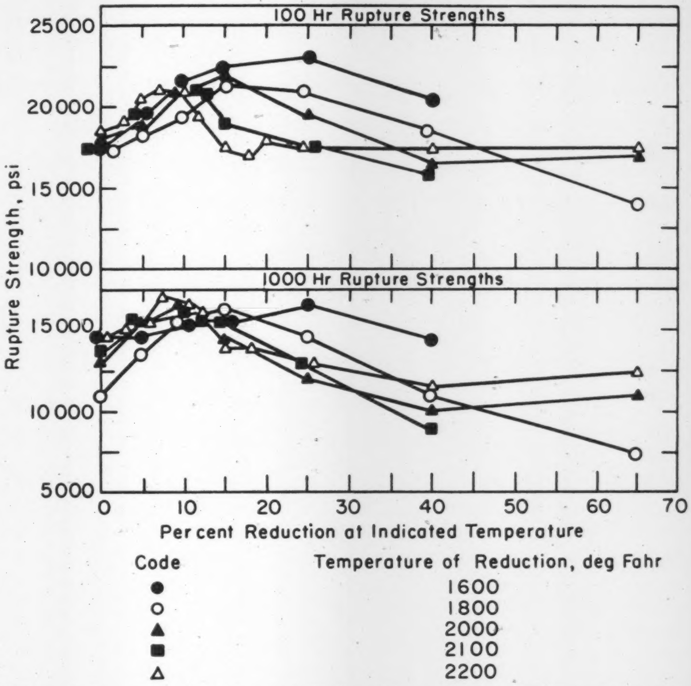


FIG. 3.—Effect of Amount of Isothermal Reduction at Various Temperatures on the 100 and 1000-hr Rupture Strengths at 1500 F.

The $\frac{7}{8}$ in. square stock was from a commerical heat having the following chemical composition:

Carbon, per cent	0.13
Manganese, per cent	1.63
Silicon, per cent	0.42
Chromium, per cent	21.22
Nickel, per cent	19.00
Cobalt, per cent	19.70
Molybdenum, per cent	2.90
Tungsten, per cent	2.61
Columbium, per cent	0.84
Nitrogen, per cent	0.13

All stock was solution-annealed at 2200 F for 1 hr, and water-quenched before heating for experimental rolling in order to minimize effects of prior commercial hot work.

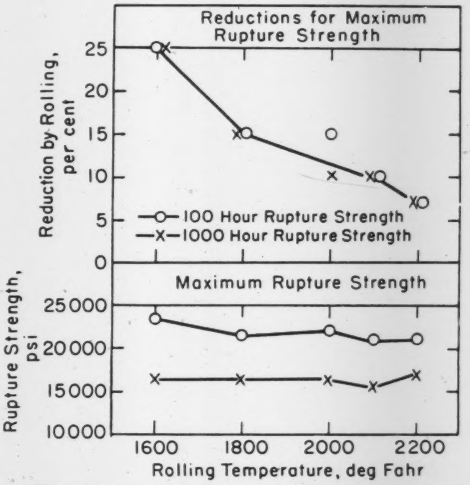


FIG. 4.—Reduction by Rolling for Maximum Rupture Strength at 1500 F.

The bar stock was rolled in open passes on a two high, non-reversible mill with 5-in. rolls. Both rolls were power driven and revolved at 70 rpm. No lubricant was used on the roll surfaces. Furnace temperatures were set 25 F above the nominal rolling temperature to average out a measured temperature drop during rolling of 50 F. The stock was air cooled after rolling. Final length of bar stock after rolling was 12 in. Rolling reductions are based upon per cent reduction for the original crosssectional area.

Rupture and creep tests at 1200 and 1500 F were used to evaluate the influence of hot working on high-temperature strength. The influence of all rolling conditions on both creep-rupture and creep strength were evaluated at 1200 and 1500 F in the hot-worked condition. Selected rolling conditions were heat treated subsequent to rolling to establish influence of hot-working on response to heat treatment. Creep-rupture tests were of sufficient duration to establish the 100 hr and 1000 hr creep-rupture strengths. The creep-rupture strengths reported were determined by double logarithmic curves of stress *versus* rupture time. The creep tests were 1000 hr in duration at 1200 and 1500 F under stresses of 25,000 and 8,000 psi, respectively. Minimum creep-rate data were established from time *versus* elongation curves obtained from both the creep tests and creep-rupture tests.

Brinell hardness numbers were used as a measure of strain hardening from hot rolling even though it was recognized that some simultaneous variations in hardness resulted from precipitation hardening. Metallographic studies of all rolled and heat treated conditions were used to evaluate structural effects. Metallographic studies were made also on creep specimens after test. Lattice parameter measurements were made because it was thought originally that variations in lattice parameter would

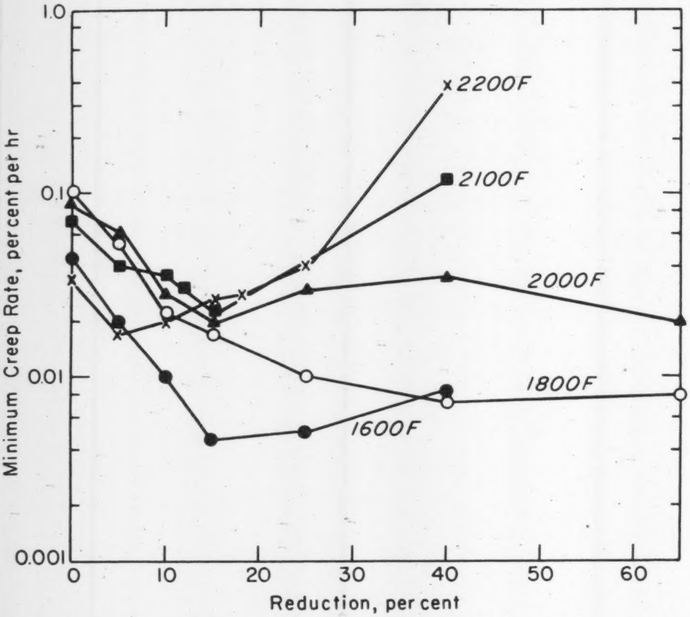


FIG. 5.—Influence of Isothermal Reductions at Indicated Temperature on the As-Rolled Minimum Creep Rates for 50,000 psi at 1200 F.

Reductions larger than 15 per cent at 1600 F, or larger than 25 per cent at 1800 F and above, required one or more reheats during rolling.

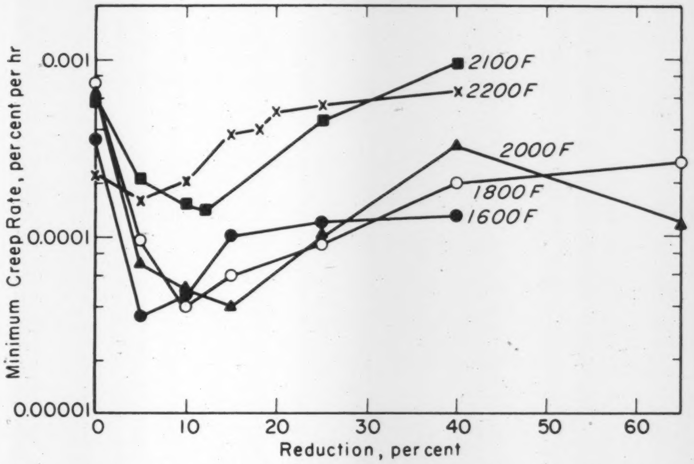


FIG. 6.—Influence of Isothermal Reductions at the Indicated Rolling Temperature on the As-Rolled Minimum Creep Rates in 1000-hr for 25,000 psi at 1200 F.

Reductions larger than 15 per cent at 1600 F or larger than 25 per cent at 1800 F and above required one or more reheats during rolling.

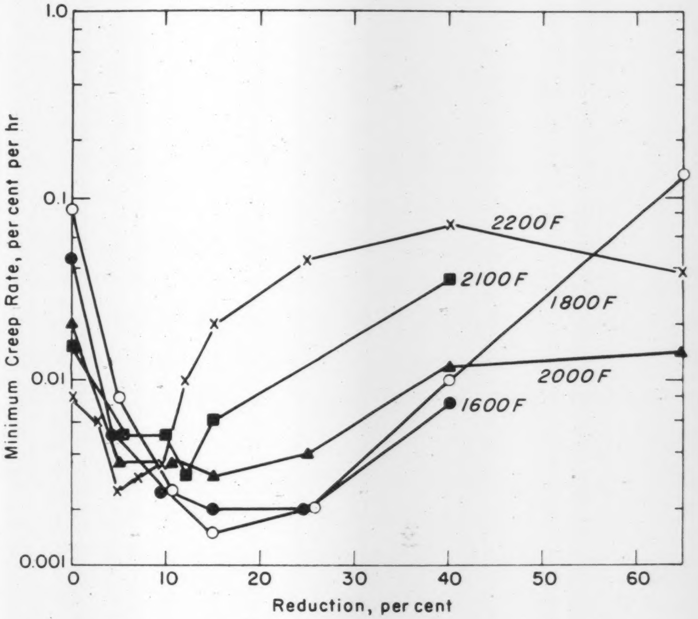


FIG. 7.—Influence of Isothermal Reductions at the Indicated Rolling Temperature on the As-Rolled Minimum Creep Rate for 15,000 psi at 1500 F.

Reductions larger than 15 per cent at 1600 F or larger than 25 per cent at 1800 F and above, required one or more reheats during rolling.

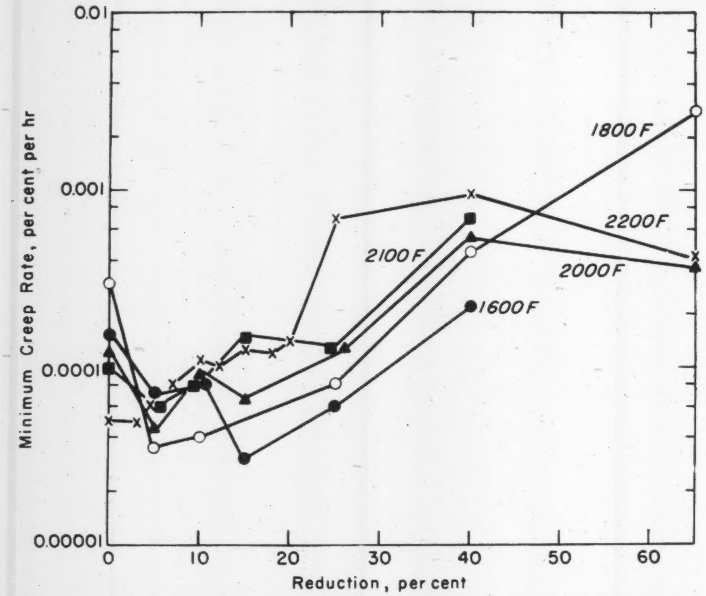


FIG. 8.—Influence of Isothermal Reductions at the Indicated Rolling Temperatures on the As-Rolled Minimum Creep Rate in 1000 hr for 8000 psi at 1500 F.

Reductions larger than 15 per cent at 1800 F and above, required one or more reheats during rolling.

reflect variations in amount of alloy constituents in solution.

RESULTS AND DISCUSSION

The influence of hot working on properties was evaluated by both creep-rupture and creep tests. Hardness tests, metallographic studies, and lattice parameter measurements were used to establish the metal structural effects. The main emphasis was placed on the properties and structural effects in the hot-worked condition. A limited study of the influence of hot working on response to heat treatment was carried out.

Attention is redirected to the fact that in all experiments the hot working was performed starting with $\frac{7}{8}$-in. square bar stock that had been heated 1 hr at 2200 F and water quenched prior to reheating for rolling. The $\frac{7}{8}$-in. starting starting stock had been commerically processed from a large arc furnace ingot.

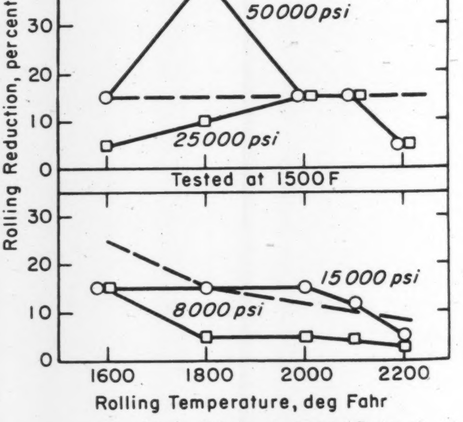


FIG. 9.—Influence of Rolling Temperature on the per cent Reduction for Optimum Creep and Rupture Properties at 1200 and 1500 F for the Indicated Conditions.

Control of Properties in the Hot-Worked Condition:

There were three outstanding results from the studies of the properties at 1200 and 1500 F in the hot-worked condition:

1. Strengths increased with amounts of hot working under isothermal conditions up to an optimum reduction (Figs. 2 through 9). Further reductions either did not improve strength or resulted in a fall-off in strength.

2. Successive reductions over a decreasing temperature range produced higher strengths at 1200 F than were obtained during working at constant temperature (Figs. 10 and 11). At 1500 F the strengths were only slightly higher than were obtained by equivalent total isothermal reductions (Figs. 10 and 12).

3. Repeated small reductions to low temperatures with reheats to below 2000 F can lead to very low creep-rupture and creep strengths (Figs. 13 and 14).

These three features of the data can be applied in a general way to account for some of the variations in strength commonly observed in this type of alloy for the hot-worked condition:

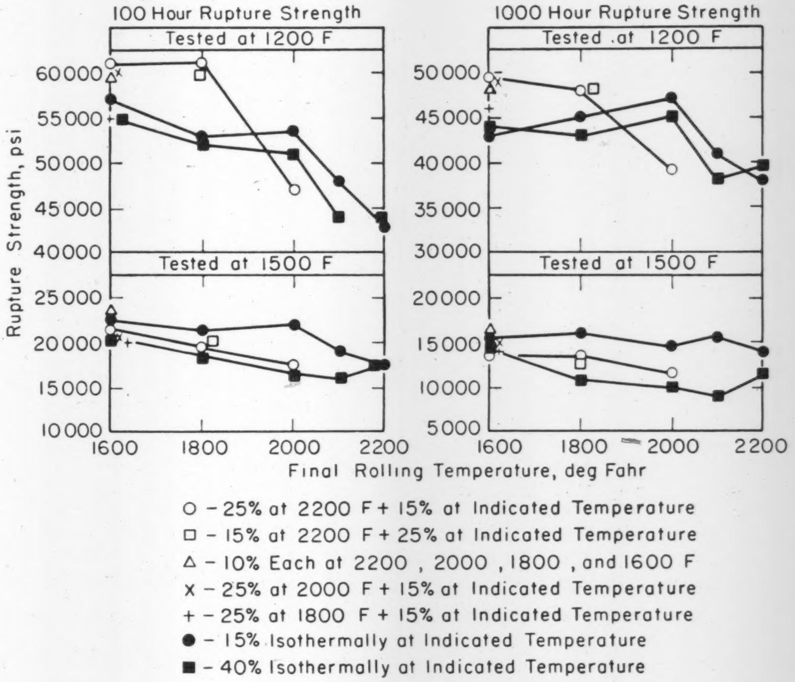


FIG. 10.—Comparison of Isothermal and Non-Isothermal Rolling on the 100 and 1000-hr Rupture Strengths at 1200 and 1500 F.

1. Medium to low strengths would be expected from large reductions at nearly constant temperature. This appears verified by being characteristic of the properties of the alloys from high volume production processes involving rapid and extensive reductions at relatively constant temperatures.

2. On the other hand, experimentally produced materials frequently have abnormally high strength in the hot-worked

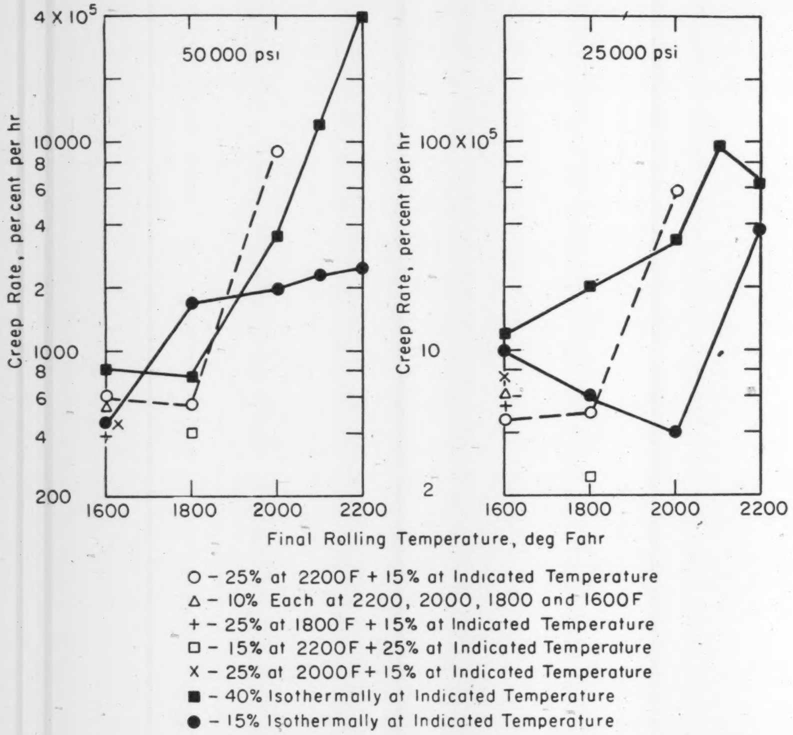


FIG. 11.—Effect of Rolling Temperature on Creep Rate at 1200 F for Various Reductions.

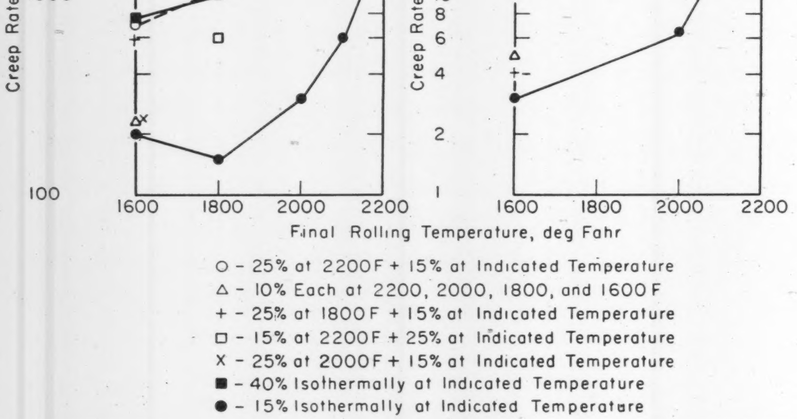


FIG. 12.—Effect of Rolling Temperature on Creep Rate at 1500 F for Various Reductions.

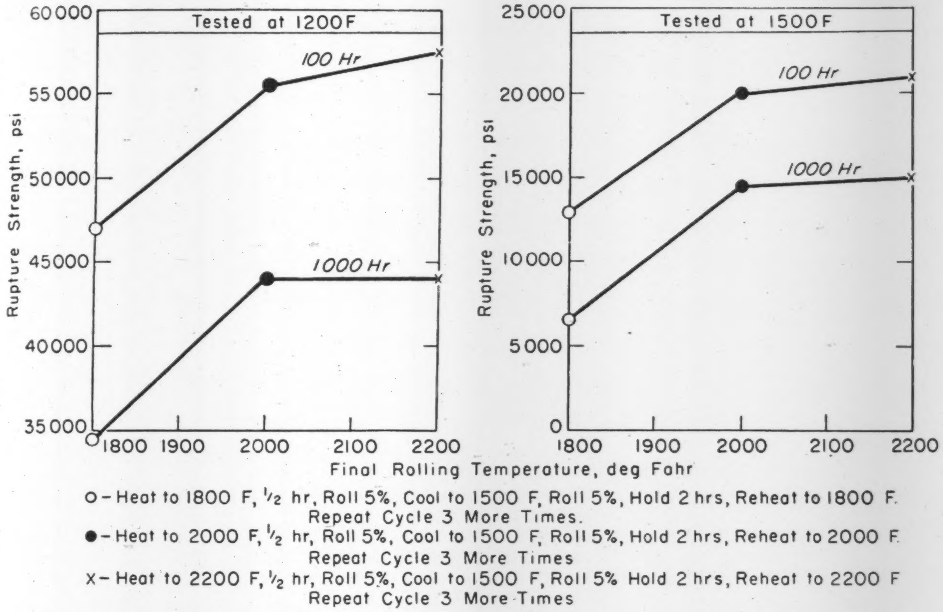


FIG. 13.—Effect of Cyclic Rolling on the 100 and 1000-hr Rupture Strengths at 1200 and 1500 F.

condition. This apparently arises from production conditions where the metal is given successive small reductions as the temperature decreases. Almost all alloys of the type studied have shown record high strengths in the hot-worked condition. A common use of small reductions with a falling temperature under these conditions seems responsible. To obtain these high strengths it appears that the working schedule must meet the following requirements:

(*a*) The reductions must either all be below the amounts causing recrystallization during working, or if recrystallization occurs at the higher temperatures, be carried down to temperatures where recrystallization ceases.

(*b*) Many small reductions at small temperature intervals are probably most effective.

The falling temperature - small reduction principle appears to have considerable importance for high strength at 1200 F. Strengths equal to or in excess of those normally obtained only by hot-cold work in the range of 1200 to 1400 F can be produced with finishing temperatures in excess of 1800 F. For example,

	Creep-Rupture Properties at 1200 F			
	100 hr		1000 hr	
	Strength, psi	Elongation, per cent	Strength, psi	Elongation, per ecnt
1. 25 per cent reduction at 2200 F + 15 per cent at 1800 F	61 000	5	48 000	6
2. Successive 10 per cent reductions at 2200, 2000, 1800 and 1600 F	60 000	20	48 000	18
3. 2050 F, 2 hr, water quenched + 15 per cent reduction at 1200 F	56 000	4	50 000	4
4. 2200 F, 1 hr, water quenched + 15 per cent reduction at 1200 F	54 000	1	52 000	5

Many small reductions at frequent temperature intervals apparently is the key to high-rupture ductility in combination with high strength (Tables I, II, and III).

3. Repeated reductions in combination with intermediate reheats to below 2000 F is undoubtedly the primary reason for low strength in sheet products when low reheat temperatures are used to reduce scaling and preserve surface quality. For the alloy studied, reheat temperatures of 2000 to 2200 F for ½ hr were adequate to avoid this and give relatively high strengths.

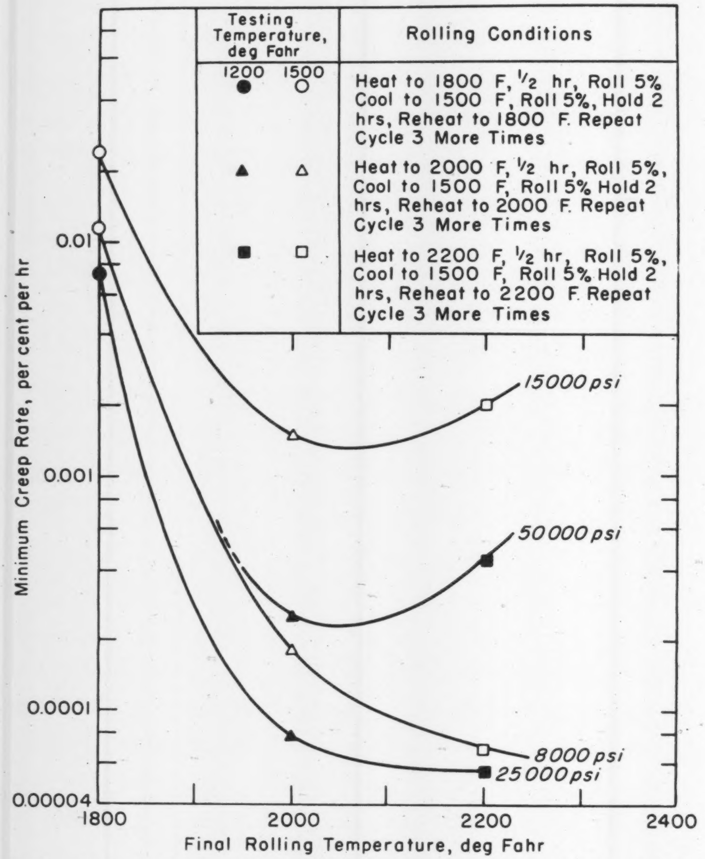


FIG. 14.—Effect of Cyclic Rolling on the Minimum Creep Rates at 1200 and 1500 F for the Indicated Initial Stresses.

In addition to the major generalities of the results there were a number of additional important features of the data of a somewhat more detailed nature relating to both mechanical and physical properties in the hot-worked condition.

1. Hardness increased with per cent reduction at all rolling temperatures. However, there was a rapid drop in hardness when the isothermal reduction exceeded 7 per cent at 2200 F and 10 per cent at 2100 F as shown in Fig. 15. Little further increase was obtained for more than 15 per cent reduction at 2000 F. All reductions at 1800 and 1600 F in-

creased hardness. In addition the over-all levels of the various hardness curves in Fig. 15 were influenced by the heating temperature alone as evidenced by the increases in hardness of stock simply reheated to the rolling temperatures and cooled without rolling. As shown in Table IV rolling over various falling temperature ranges developed high as-rolled hardness values, the one exception being the material rolled between 2200 and 2000 F. Cyclic rolling between 1500 and 1800 F, and between 1500 and 2000 or 2200 F, developed hardnesses of 253 and 248 BHN, respectively.

2. The microstructural studies of the as-rolled specimens (Figs. 16, 17 and 18) showed that the discontinuities in the per cent reduction *versus* hardness curves (Fig. 15) were due to recrystallization during rolling. Thus recrystallization during hot working is dependent upon the temperature and amount of reduction. Even at 2200 F a reduction of 7 per cent was required to initiate recrystallization. Reductions of between 15 and 25 per cent at 2000 F were required before partial recrystallization occurred. Further reduction at this temperature increased the amount of recrystallization with little change in hardness. Grain distortion rather than recrystallization was observed for rolling at 1800 or 1600 F.

Rolling first at 2200 F before rolling at lower temperatures, Fig. 17, reduced the grain size by recrystallization during the initial working at 2200 F.

3. A finely dispersed matrix precipitate formed when the alloy was reheated to 1800 or 2000 F for ½ hr. Conceivably this high-temperature precipitate, spheroidized and agglomerated, is the source of the excess phases other than the insoluble columbium compounds, frequently observed in the structure of alloys of the type being considered.

Rolling first at 2200 F before rolling at 2000 or 1800 F, Fig. 17, suppressed

TABLE I.—RUPTURE DUCTILITY AT 1200 AND 1500 F FOR BAR STOCK ROLLED ISOTHERMALLY BETWEEN 1600 AND 2200 F.

Rolling Condition	Interpolated Rupture Elongation at 1200 F, per cent in 1 in.		Interpolated Rupture Elongation at 1500 F, per cent in 1 in.	
	100 hr	1000 hr	100 hr	1000 hr
Rolled at 1600 F, reduction				
0 per cent....	9	7	49	...
5 per cent....	5	8	37	25*
10 per cent....	3	3	30	20
15 per cent....	5	5	14	6
25 per cent....	3	5	5	6
40 per cent....	4	4	12	8*
Rolled at 1800 F, reduction				
0 per cent....	10	20	57	26
5 per cent....	10	9	55	23
10 per cent....	8	20	40	15
15 per cent....	6	15	35	5
25 per cent....	4	9	10	5*
40 per cent....	6	5	4	7
65 per cent....	20	23	25	20
Rolled at 2000 F, reduction				
0 per cent....	10	14	60	32
5 per cent....	6	8*	35	19
10 per cent....	5	12	20	9
15 per cent....	6	16	17	5
25 per cent....	9	11	12	6
40 per cent....	20	19	17	9
65 per cent....	...	...	22	15
Rolled at 2100 F, reduction				
0 per cent....	5	...	40	35*
5 per cent....	6	12	35	20
10 per cent....	11	11	30	15*
12 per cent....	9	15	22	10
15 per cent....	13	15*	20	6
25 per cent....	...	...	25	10*
40 per cent....	11	17	50	25
Rolled at 2200 F, reduction				
0 per cent....	5	7	18	25
3 per cent....	...	...	35	41
5 per cent....	8	6	25	13*
7 per cent....	8	...	24	12*
10 per cent....	7	5	21	12
12 per cent....	7	8	23	10
15 per cent....	9	12	19	10
18 per cent....	7	19	24	15
20 per cent....	...	...	...	...
25 per cent....	6	6	40	30
40 per cent....	9	15	44	32
65 per cent....	...	...	50	35

* Extrapolated.

TABLE II.—RUPTURE DUCTILITY AT 1200 AND 1500 F FOR BAR STOCK ROLLED OVER CONTROLLED TEMPERATURE RANGES.

Rolling Condition	Interpolated Rupture Elongation at 1200 F, per cent in 1 in.		Interpolated Rupture Elongation at 1500 F, per cent in 1 in.	
	100 hr	1000 hr	100 hr	1000 hr
Rolled 25 per cent at 2200 F plus 15 per cent at 2000 F	10	20	30	20
Rolled 25 per cent at 2200 F plus 15 per cent at 1800 F	5	6	15	...
Rolled 15 per cent at 2200 F plus 25 per cent at 1800 F	5	8	5	5
Rolled 25 per cent at 2200 F plus 15 per cent at 1600 F	3	3*	19	5
Rolled 10 per cent each at 2200, 2000, 1800, and 1600 F	20	18	28	16
Rolled 25 per cent at 2000 F plus 15 per cent at 1600 F	10	...	3	5*
Rolled 25 per cent at 1800 F plus 15 per cent at 1600 F	5	...	5	...

* Extrapolated.

TABLE III.—RUPTURE DUCTILITY AT 1200 AND 1500 F FOR CYCLIC ROLLED BAR STOCK.

Rolling Condition	Interpolated Rupture Elongation at 1200 F, per cent in 1 in.		Interpolated Rupture Elongation at 1500 F, per cent in 1 in.	
	100 hr	1000 hr	100 hr	1000 hr
Heat to 1800 F, ½ hr, roll 5 per cent, cool to 1500 F, roll 5 per cent, hold 2 hr, reheat to 1800 F. Repeat cycle 3 more times	40	30	20	10
Heat to 2000 F, ½ hr, roll 5 per cent, cool to 1500 F, roll 5 per cent, hold 2 hr, reheat to 1800 F. Repeat cycle 3 more times	18	15	12	5
Heat to 2200 F, ½ hr, roll 5 per cent, cool to 1500 F, roll 5 per cent, hold 2 hr, reheat to 1800 F. Repeat cycle 3 more times	20	20	25	14

the high-temperature precipitation. Rolling at 1600 F did not produce a general precipitate. Repeated rolling at 1500 and 1800 F caused extensive precipitation and agglomeration of the precipitates. There was little evidence of this when the upper temperature was increased to 2000 or 2200 F (Fig. 18).

Structural changes during testing at 1200 F were largely dependent on the initial as-rolled condition of the bar stock. Extensive precipitation took place in the matrix during testing provided precipitation had occurred during rolling (Fig. 19). The structural changes which occurred during testing at 1500 F appeared to be largely independent of the initial conditions of the microstructure. Precipitation or agglomeration occurred in all bars during testing and all structures were remarkably similar after testing (Fig. 20).

4. The influence of amount and temperature of reduction on lattice parameters (Figs. 21, 22 and 23) was complex. Successive minimum and maximum

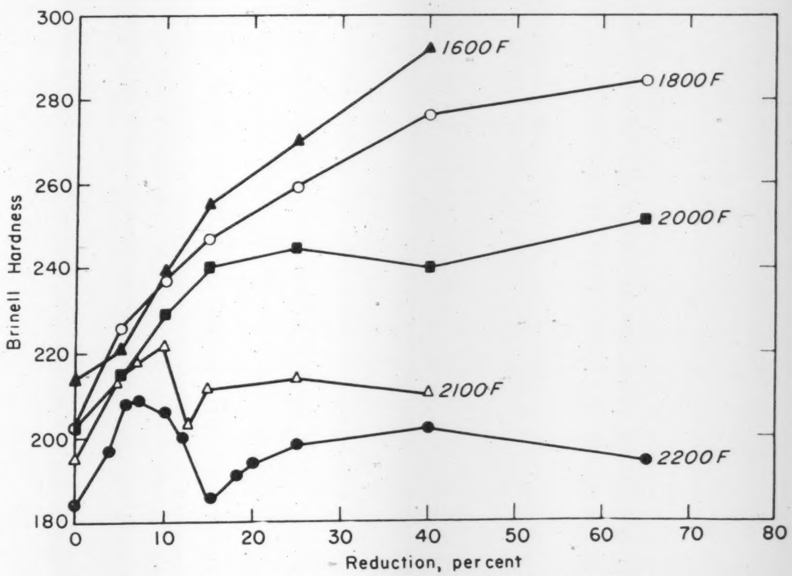


FIG. 15.—Influence of Isothermal Reductions at 1600, 1800, 2000, 2100, or 2200 F on the As-Rolled Hardness.

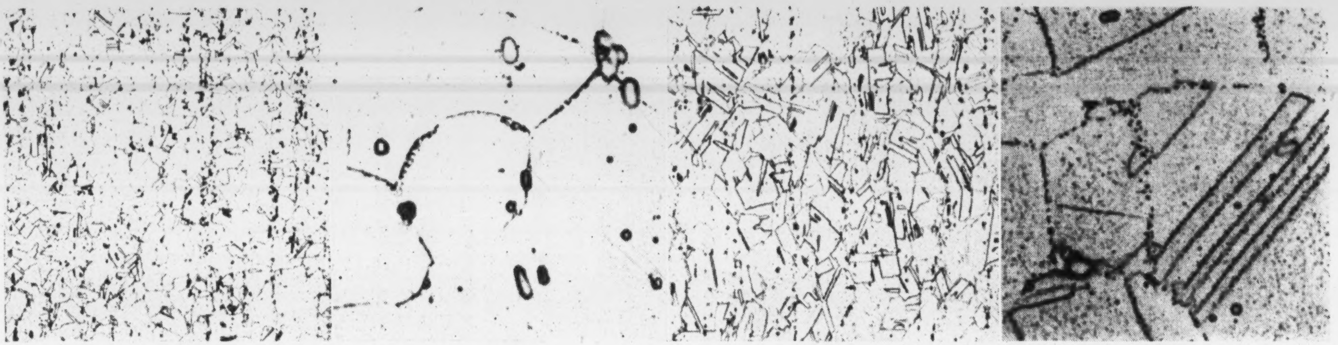
Reductions larger than 15 per cent at 1600 F, or larger than 25 per cent at 1800 F and above, required one or more reheats during rolling.

values appeared as amount of reduction was increased. The amount of reduction required to produce these effects increased as the rolling temperature was reduced. The agreement between measurements made transverse to the rolling direction with check determinations at other angles (Fig. 21) indicates that any orientation effects were small. During the investigation it was established that cooling rate had a pronounced effect (Figs. 24 and 25) on measured lattice parameter. These limited data show that intermediate cooling rates result in larger parameters. That is, air cooling produced larger values than either very slow or very rapid cooling.

5. The maximum creep-rupture strength at 1200 F from isothermal rolling was obtained by 15 per cent reduction at any temperature (Fig. 2). The hot-worked condition generally yields creep-rupture strengths at 1200 F which are higher than can be obtained by heat treatment alone. The tempera-

TABLE IV.—BRINELL HARDNESS OF BAR STOCK ROLLED OVER CONTROLLED TEMPERATURE RANGES.

Rolling Conditions	Brinell Hardness, 3000 kg load
25 per cent at 2200 F plus 15 per cent at 2000 F	221
25 per cent at 2200 F plus 15 per cent at 1800 F	272
15 per cent at 2200 F plus 25 per cent at 1800 F	273
25 per cent at 2200 F plus 15 per cent at 1600 F	278
10 per cent each at 2200, 2000, 1800, and 1600 F	274
25 per cent at 2000 F plus 15 per cent at 1600 F	283
25 per cent at 1800 F plus 15 per cent at 1600 F	283
Heat to 1800 F, ½ hr., roll 5 per cent, cool to 1500 F, roll 5 per cent, hold 2 hr., reheat to 1800 F. Repeat cycle 3 more times	253
Heat to 2000 F, ½ hr., roll 5 per cent, cool to 1500 F, roll 5 per cent, hold 2 hr., reheat to 2000 F. Repeat cycle 3 more times	248
Heat to 2200 F, ½ hr., roll 5 per cent, cool to 1500 F, roll 5 per cent, hold 2 hr., reheat to 2200 F. Repeat cycle 3 more times	248

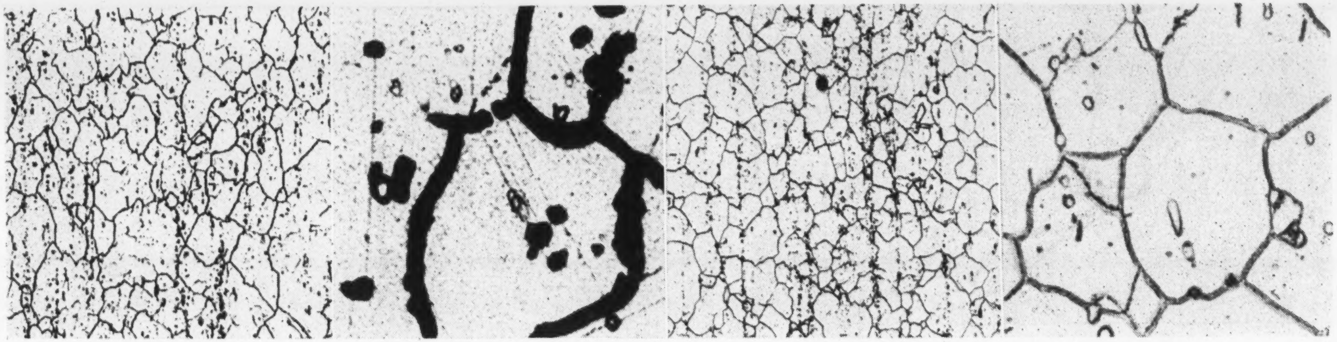


×100 ×1000

Rolled 15 per cent at 2200 F.

×100 ×1000

Rolled 15 per cent at 2000 F.



×100 ×1000

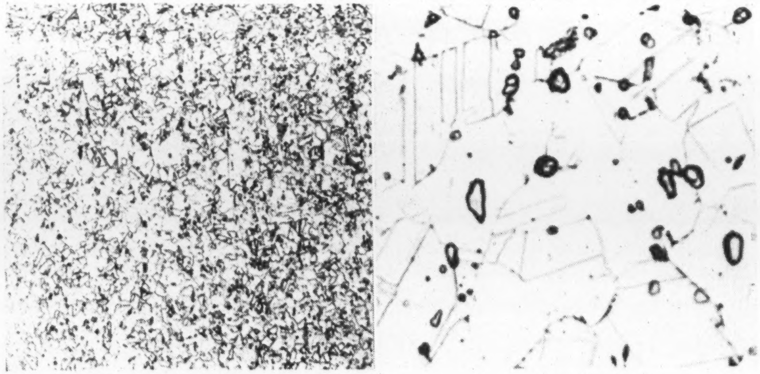
Rolled 15 per cent at 1800 F.

×100 ×1000

Rolled 15 per cent at 1600 F.

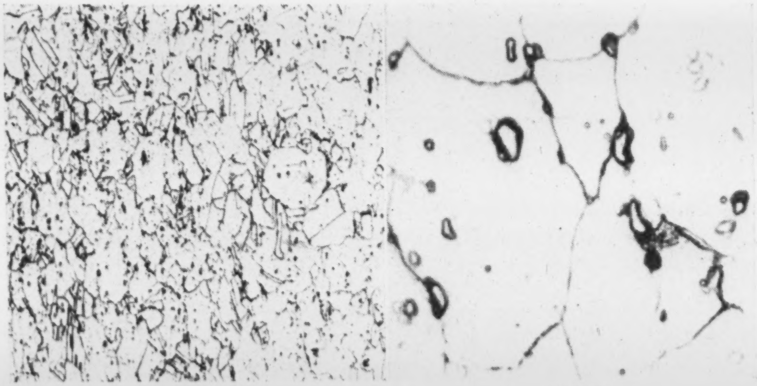
FIG. 16.—Effect of 15 per cent Isothermal Reduction at Temperatures Indicated on Microstructure.

Bar stock was solution treated at 2200 F, 1 hr, and water quenched prior to rolling. (Electrolytically etched in 10 per cent chromic acid.)



×100 ×1000

Rolled 25 per cent at 2200 F plus 15 per cent at 2000 F.

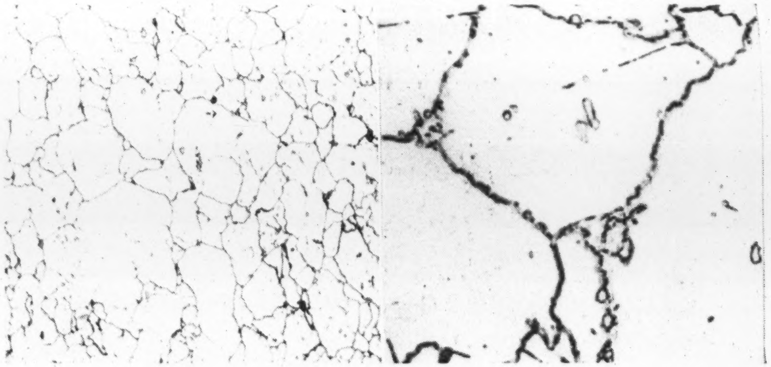


×100 ×1000

Rolled 10 per cent each at 2200, 2000, 1800 and 1600 F.

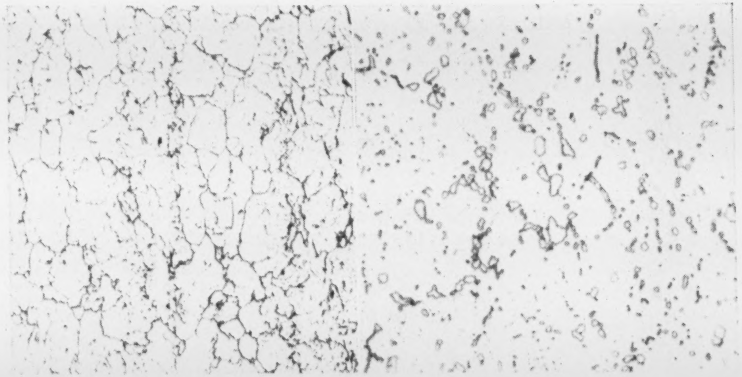
FIG. 17.—Effects of Non-Isothermal Reductions on Microstructure.

Bar stock was solution treated at 2200 F, 1 hr, water quenched and then rolled as indicated. (Electrolytically etched in 10 per cent chromic acid.)



×100 ×1000

Heated to 2200 F, ½ hr, rolled 5 per cent, cooled to 1500 F, 2 hr, reheated to 2200 F. Repeated cycle three more times

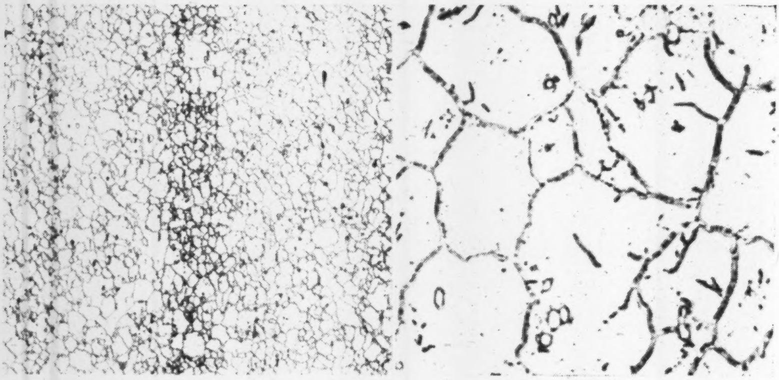


×100 ×1000

Heated to 1800 F, ½ hr., rolled 5 per cent, cooled to 1500 F, rolled 5 per cent, held 2 hr, reheated to 1800 F. Repeated cycle three more times.

FIG. 18.—Effect of Cyclic Rolling on the Microstructures.

Bar stock was solution treated at 2200 F, 1 hr, water quenched and rolled as indicated. (Electrolytically etched in 10 per cent chromic acid.)

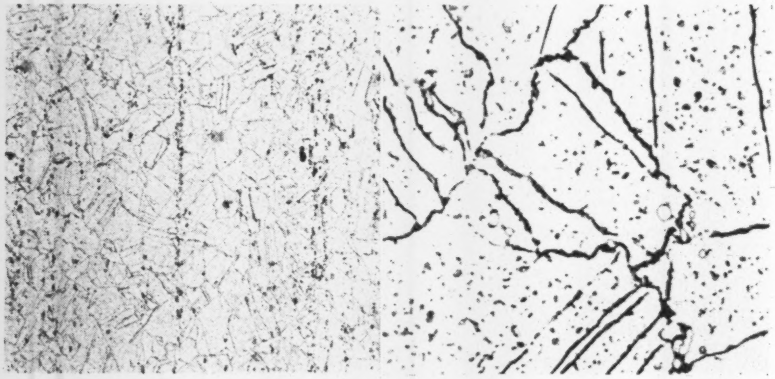


×100 ×1000

Rolled 25 per cent at 2200 F plus 15 per cent at 2000 F. Duration of Creep Test: 1125 hr.

FIG. 19.—Microstructure After Creep Testing at 1200 F with a Stress of 25,000 psi.

Prior to testing, bar stock was solution treated at 2200 F, 1 hr, water quenched and rolled as indicated. (Electrolytically etched in 10 per cent chromic acid.)



×100 ×1000

Rolled 15 per cent at 2000 F. Duration of Creep Test: 1154 hr.

FIG. 20.—Typical Microstructure After Creep Testing at 1500 F with a Stress of 25,000 psi.

Prior to testing, bar stock was solution treated at 2200 F, 1 hr, water quenched and rolled as indicated. (Electrolytically etched in 10 per cent chromic acid.)

ture of working has a potent influence on the level of creep-rupture strength at 1200 F (Fig. 2). Relatively high creep-rupture strengths for isothermally reduced stock in excess of 50,000 and 40,000 psi for 100 and 1000 hr, respectively, require working below 2100 F.

6. The control of rupture strength at 1500 F in the isothermally hot-worked condition is dependent mostly on the degree of reduction and only slightly dependent upon the working temperature (Fig. 3). Specific reductions dependent on the temperature of working (Fig. 4) are required for maximum strength with large reductions being detrimental. It is noteworthy that a 7 per cent reduction at 2200 F yielded as high a rupture strength at 1500 F as could be obtained by any other conditions of working investigated.

7. Apparently high elongation and reduction of area in rupture tests at 1200 F is dependent on large reductions

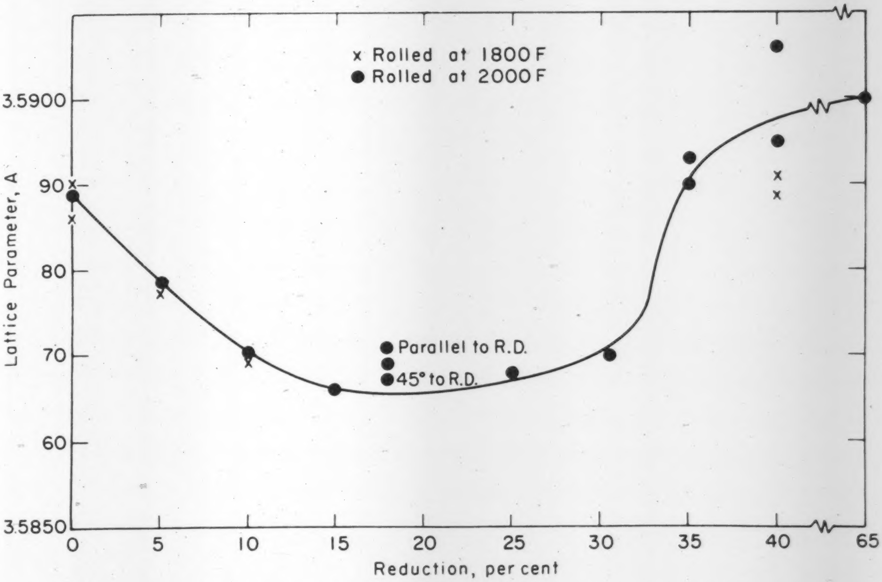


FIG. 21.—Influence of Isothermal Reductions at 1800 or 2000 F on the Lattice Parameter of As-Rolled Bar Stock.

Reductions larger than 25 per cent required one or more reheats during rolling. All specimens are transverse to rolling direction unless indicated otherwise.

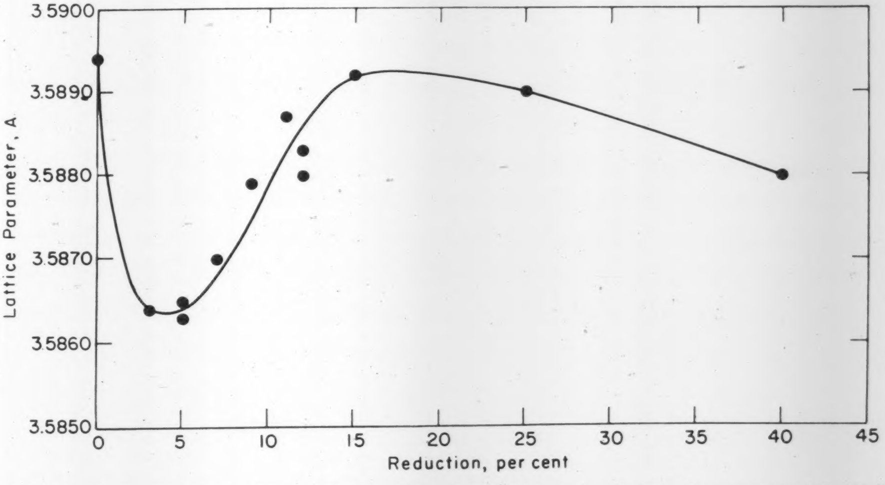


FIG. 22.—Influence of Isothermal Reductions at 2100 F on the Lattice Parameter of As-Rolled Bar Stock.

The reduction of 40 per cent required one reheat during rolling.

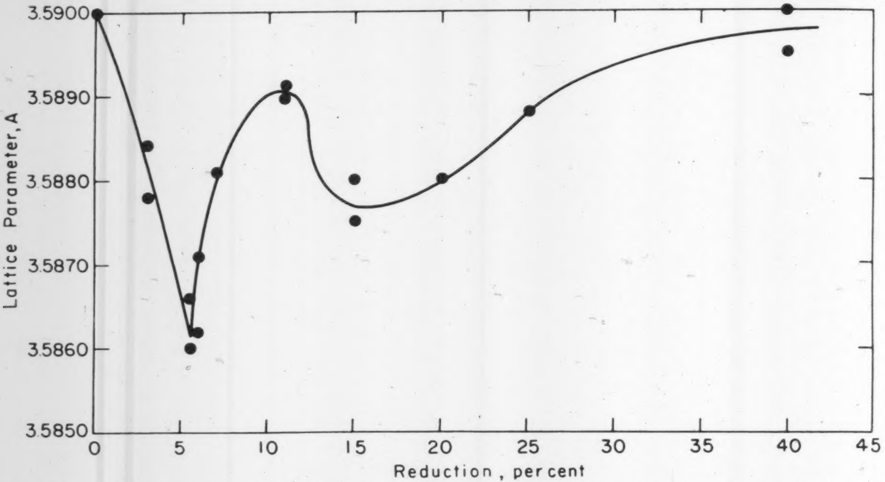


FIG. 23.—Influence of Isothermal Reductions at 2200 F on the Lattice Parameter of As-Rolled Bar Stock.

The reduction of 40 per cent required one reheat during rolling.

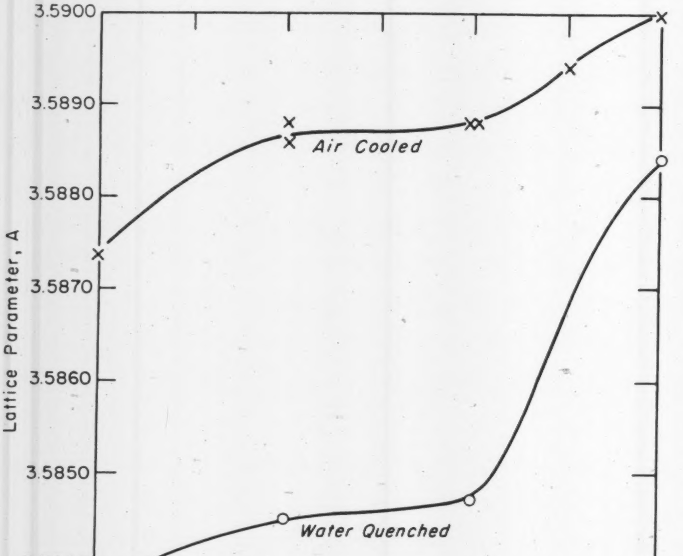


FIG. 24.—Influence of Cooling Rate from Reheat Temperature on Lattice Parameter.

Specimens solution treated at 2200 F, 1 hr, water quenched, reheated to indicated reheat temperature for ½ hr, and cooled as indicated.

at 1800 to 2000 F (Tables I and II). High-temperature working with recrystallization also increased ductility.

Elongation and reduction of area in rupture tests at 1500 F was very sensitive to reduction amounts (Tables I and II). Ductility can be reduced to very low values by increasing amounts of reduction. For isothermal working high ductility is obtained if temperature of working exceeds 2000 F or if the reductions are very small.

8. Creep resistance at low stress levels is apparently more sensitive to degree of reductions than rupture strength. (Compare Figs. 6 and 8 with 2 and 3.) At 1200 F, the creep strengths were sensitive to temperatures of hot working just as the creep-rupture strengths were sensitive (Fig. 6). Large reductions above 2000 F develop low creep resistance. At 1500 F, creep resistance was more sensitive to amount of reduction (Fig. 8). The data indicate that large reductions below 2000 F might be particularly damaging.

9. Generally speaking the optimum reduction for maximum creep resistance under low stresses is less than for maximum rupture strength (Fig. 9).

10. Recrystallization during working without further working at lower temperatures leads to low hardness and relatively low strength (Tables I and II).

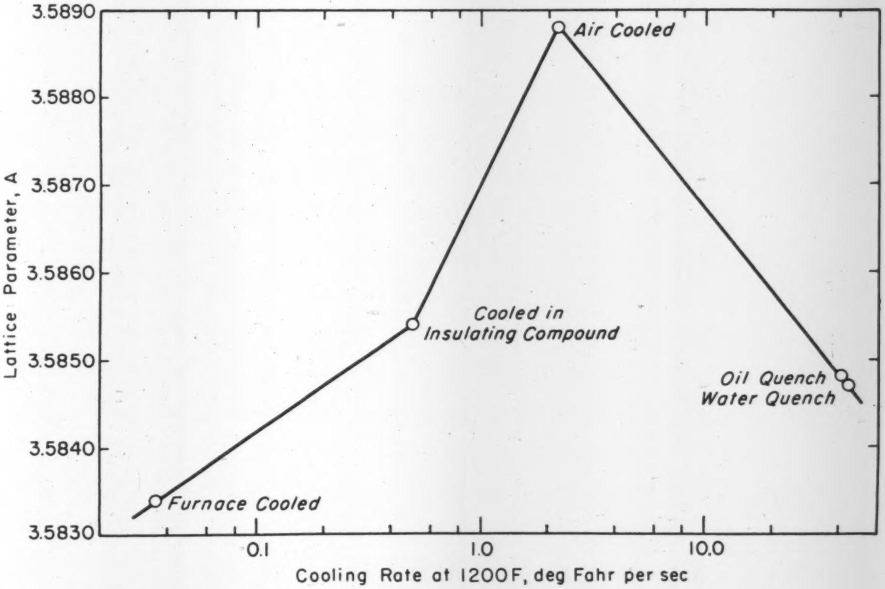


FIG. 25.—Influence of Cooling Rate from 2025 F on the Lattice Parameter.

After solution treating at 2200 F, 1 hr, water quenched, specimens were reheated to 2025 F, ½ hr, and cooled as indicated.

Mechanism of Strengthening and Weakening by Hot-Working:

The results of this investigation provide a basis for hypothesis to explain the observed influences of hot working on the high-temperature strength properties of this class of alloys. Conceivably both strengthening and weakening occur during working as evidenced by increases in strength followed by decreases as the amount of hot working increases. The relative effects vary with stress and

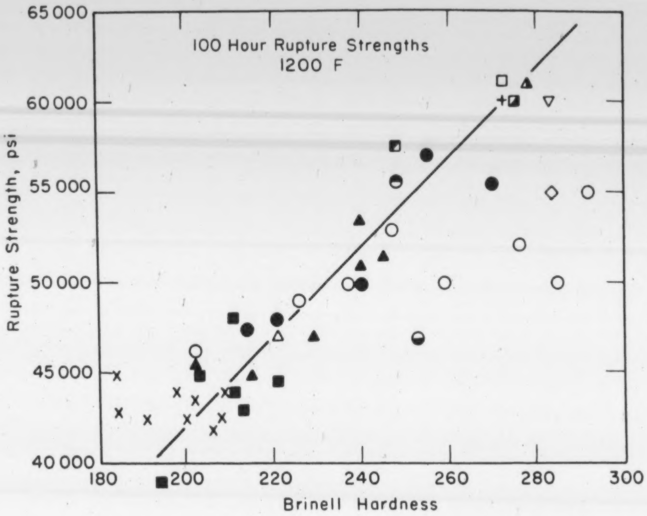


FIG. 26.—Correlation of the 100-hr Rupture Strength at 1200 F with the As-Rolled Brinell Hardness.

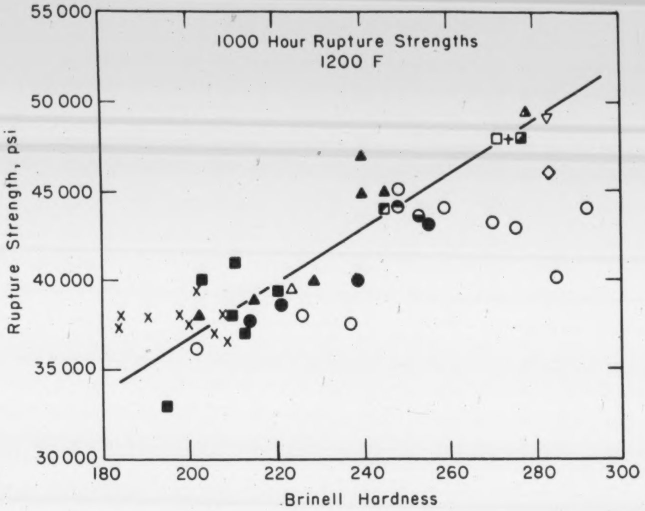
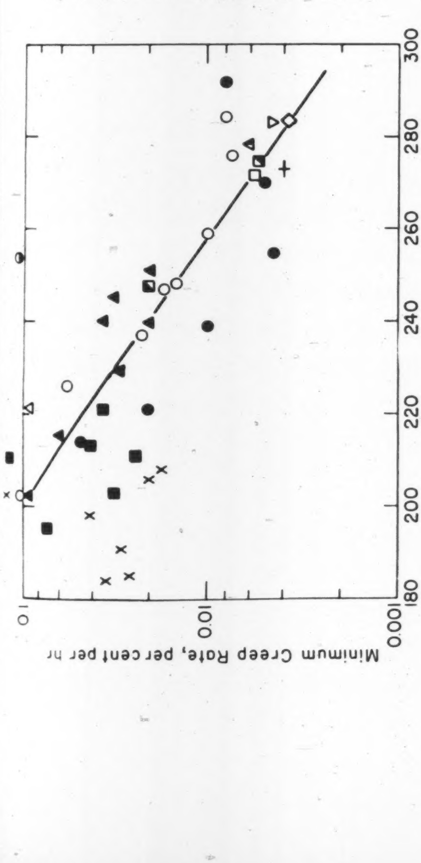


FIG. 27.—Correlation of the 1000-hr Rupture Strength at 1200 F with the As-Rolled Brinell Hardness.

X - Rolled at 2200 F
■ - Rolled at 2100 F
● - Rolled at 1600 F
▲ - Rolled at 2200 F
○ - Rolled at 1800 F

△ - 25 Per cent at 2200 F Plus 15 per cent at 2000 F
□ - 25 Per cent at 2200 F Plus 15 per cent at 1800 F
+ - 15 Per cent at 2200 F Plus 25 per cent at 1800 F
▲ - 25 Per cent at 2200 F Plus 15 per cent at 1600 F
◪ - 10 Per cent Each at 2200 , 2000 , 1800 , and 1600 F
▽ - 25 Per cent at 2000 F Plus 15 per cent at 1600 F
◇ - 25 Per cent at 1800 F Plus 15 per cent at 1600 F
◒ - Heat to 1800 F, ½ hr., Roll 5%, Cool to 1500 F, Roll 5%, Hold 2 hrs., Reheat to 1800 F. Repeat Cycle 4 Times.
◓ - Heat to 2000 F., ½ hr., Roll 5%, Cool to 1500 F, Roll 5%, Hold 2 hrs., Reheat to 2000 F. Repeat Cycle 4 Times.
◩ - Heat to 2200 F, ½ hr., Roll 5%, Cool to 1500 F, Roll 5%, Hold 2 hrs., Reheat to 2000 F. Repeat Cycle 4 Times.



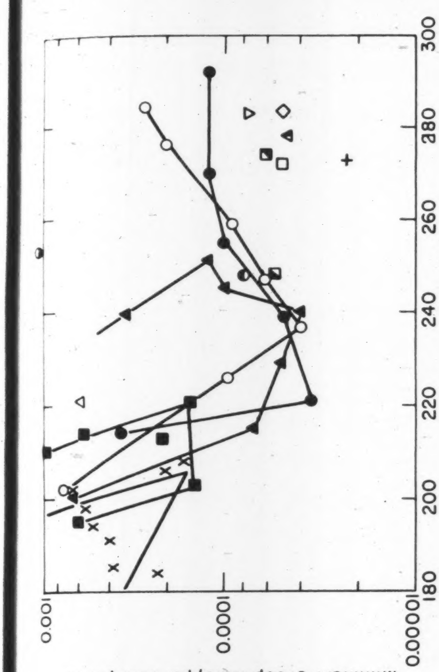
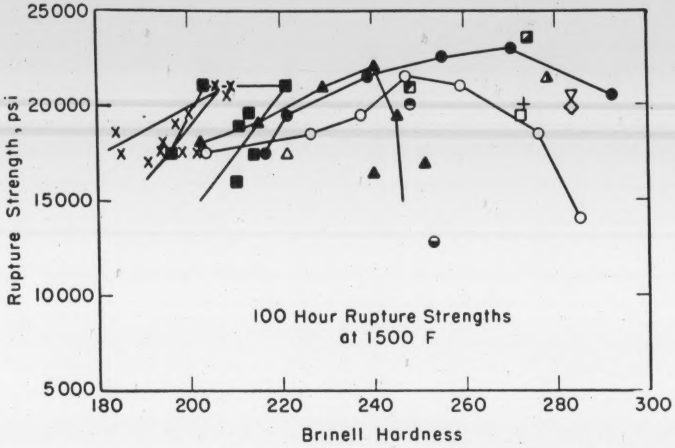
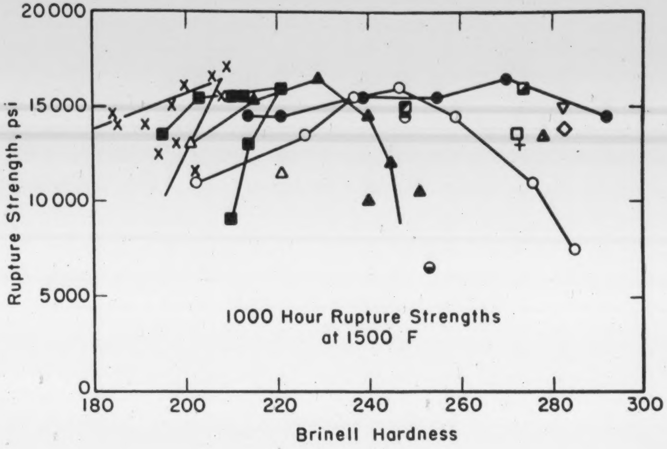


FIG. 28.—Correlation of Minimum Creep Rate for an Initial Stress of 50,000 psi at 1200 F with the As-Rolled Brinell Hardness.

FIG. 29.—Correlation of Minimum Creep Rate in a 1000 hr for an Initial Stress of 25,000 psi at 1200 F with the As-Rolled Brinell Hardness.



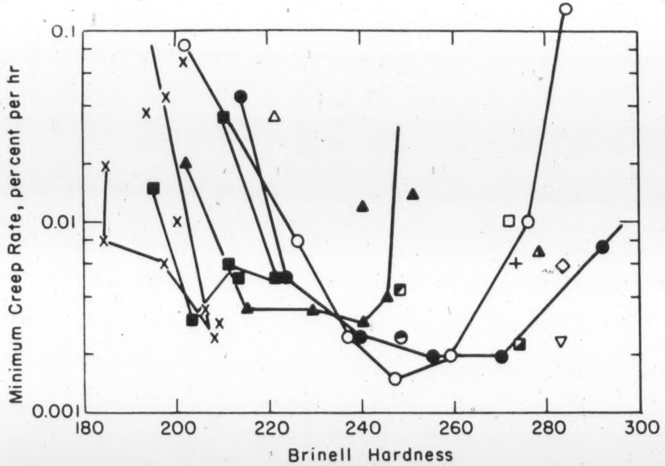


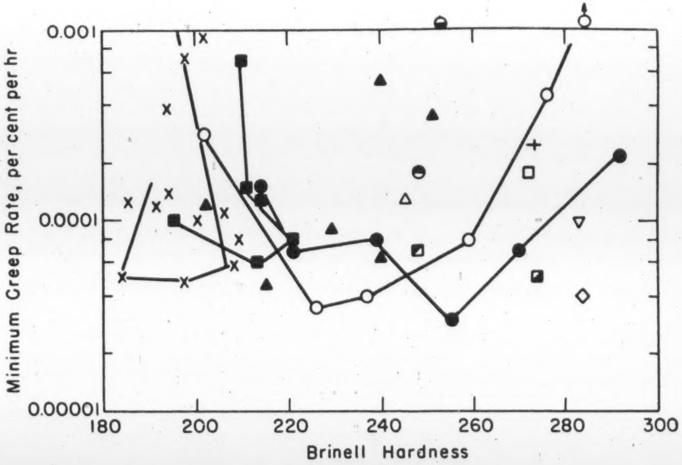
X - Rolled at 2200 F
■ - Rolled at 2100 F
▲ - Rolled at 2000 F
○ - Rolled at 1800 F
● - Rolled at 1600 F

△ - 25 Per cent at 2200 F Plus 15 per cent at 2000 F
□ - 25 Per cent at 2200 F Plus 15 per cent at 1800 F
+ - 15 Per cent at 2200 F Plus 25 per cent at 1800 F
▲ - 25 Per cent at 2200 F Plus 15 per cent at 1600 F
◪ - 10 Per cent Each at 2200, 2000, 1800, and 1600 F
▽ - 25 Per cent at 2000 F Plus 15 per cent at 1600 F
◇ - 25 Per cent at 1800 F Plus 15 per cent at 1600 F
◓ - Heat to 1800 F, 1/2 hr, Roll 5%, Cool to 1500 F, Roll 5%, Hold 2 hrs, Reheat to 1800 F Repeat Cycle 4 Times.
◒ - Heat to 2000 F, 1/2 hr, Roll 5%, Cool to 1500 F, Roll 5%, Hold 2 hrs, Reheat to 2000 F Repeat Cycle 4 Times.
◩ - Heat to 2200 F, 1/2 hr, Roll 5%, Cool to 1500 F, Roll 5%, Hold 2 hrs, Reheat to 2000 F Repeat Cycle 4 Times.

FIG. 30.—Correlation of the 100-hr Rupture Strengths at 1500 F with the As-Rolled Brinell Hardness.

FIG. 31.—Correlation of the 1000-hr Rupture Strength at 1500 F with the As-Rolled Brinell Hardness.





X - Rolled at 2200 F
■ - Rolled at 2100 F
▲ - Rolled at 2000 F
○ - Rolled at 1800 F
● Rolled at 1600 F

△ - 25 Per cent at 2200 F Plus 15 per cent at 2000 F
□ - 25 Per cent at 2200 F Plus 15 per cent at 1800 F
+ - 15 Per cent at 2200 F Plus 25 per cent at 1800 F
▲ - 25 Per cent at 2200 F Plus 15 per cent at 1600 F
◪ - 10 Per cent Each at 2200 , 2000 , 1800 , and 1600 F
▽ - 25 Per cent at 2000 F Plus 15 per cent at 1600 F
◇ - 25 Per cent at 1800 F Plus 15 per cent at 1600 F
◒ - Heat to 1800 F, 1/2 hr, Roll 5%, Cool to 1500 F, Roll 5%, Hold 2 hrs, Reheat to 1800 F Repeat Cycle 4 Times.
◓ - Heat to 2000 F, 1/2 hr, Roll 5%, Cool to 1500 F, Roll 5%, Hold 2 hrs, Reheat to 2000 F Repeat Cycle 4 Times.
◩ - Heat to 2200 F, 1/2 hr, Roll 5%, Cool to 1500 F, Roll 5%, Hold 2 hrs, Reheat to 2200 F Repeat Cycle 4 Times.

FIG. 32.—Correlation of Minimum Creep Rate for an Initial Stress of 15,000 psi at 1500 F with the As-Rolled Brinell Hardness.

FIG. 33.—Correlation of Minimum Creep Rate in 1000 hr for an Initial Stress of 8000 psi at 1500 F with the As-Rolled Brinell Hardness.

temperature of testing. It appears that a major factor in strengthening involves strain hardening, although this undoubtedly is an oversimplification. The suggestion is made that weakening originates mainly from a recovery type process during working which in its extreme exhibits itself as recrystallization during working at the higher temperatures. When recrystallization does not actually occur, the damage arises from the same structural alterations as those which induce recrystallization to occur at higher temperatures. In addition, there are other effects from the precipitation during working at 1600 to 2000 F and during working.

Strengthening During Working.—The correlations of hardness to rupture and creep strengths (Figs. 26 through 33) show reasonably close relationships between hardness and rupture strengths at 1200 F. When the stress was reduced to 25,000 psi at 1200 F, the resulting creep strength did not correlate as well. Both rupture and creep strengths at 1500 F correlated poorly with hardness. Even though it is recognized that hardness is an imperfect indication of strain hardening, it is suggested that the correlation at 1200 F for rupture and creep at high stress levels presents good evidence that when creep is largely a slip process under relatively low-temperature rapid creep conditions, strain hardening is a major controlling factor. As the creep rate is reduced or the test temperature increased the creep process becomes more "viscous" in nature. Strain hardening then becomes less effective and the correlation breaks down.

Weakening During Working.—The appearance of recrystallization must be a definite limitation to strengthening from hot working. Strengths did not increase with reductions at 2200 F, presumably due to continuous recrystallization during rolling. Continuous recrystallization during working first at 2200 and then at 2000 F was accompanied also by low strength.

Recrystallization is a recovery process and appears first in the grain boundaries. Larger reductions result in its initiation within grains. The suggestion is made that in the absence of recrystallization the same structural alterations which lead to recrystallization also lower creep resistance as creep becomes more a function of grain boundary conditions (lower creep rates or higher temperatures), and probably accumulated damage within crystals. Since visual recrystallization causes weakening, or damage, it seems reasonable to presume that some type of recovery or similar process such as subgrain formation occurring prior to, or in the absence of recrystallization, can cause damage. Such a damage component is cumulative, apparently because rupture strengths at 1500 F and creep resistance at low stress levels at both 1200 and 1500 F was reduced increasingly as reductions were increased past the optimum. Moreover, it appears at smaller reductions as the creep stress is reduced and the test temperature increased (Fig. 9), as such a theory would predict.

In fact, due to the analogy of increasing damage from increasing reduction as creep becomes more viscous in nature, there is reason to suspect that a major source of damage may be the non-slip or viscous flow so long identified by investigators with rapid plastic deformation. Certainly plastic deformation is non-homogeneous in polycrystalline aggregates and gives evidence of both slip and non-slip processes.

Detailed Experimental Results Relating to Mechanism.—The optimum reduction during isothermal working for maximum rupture strength at 1200 F was constant at 15 per cent. This suggests that the damage component begins to predominate at this reduction regardless of the temperature of working. There is in fact considerable reason to believe that 15 per cent reduction gives optimum strength for temperatures of reduction as low as 1000 F when the stock is initially solution treated at 2200 F (**1**)[3]. Apparently hardness can continue to increase with further reduction in the absence of recrystallization, but the rupture strength cannot. This results in strengths no longer correlating with hardness (Figs. 26 and 27) when worked at 1800 and 1600 F and probably at lower temperatures. It was shown by Frey, *et al* (**2**) that correlation with internal strain broke down for creep resistance at 1200 F under 50,000 psi for a 40 per cent reduction at 76 F. It now seems, however, that this breakdown was due to excessive deformation rather than to recovery during testing as proposed originally.

To account for observed behavior, it seems necessary to postulate that only strain-hardening accumulated with reductions up to 15 per cent at any temperature is effective before the damage component prevents further strengthening from increasing strain hardening. It would certainly be easiest to explain this if subgrain formation controlled rupture strength and was dependent largely on degree of reduction and independent of working temperature. This explanation would best require a rupture strength independent of working temperature. Actually, this is not far from the facts. In Fig. 34, rupture data for 15 per cent reductions down to 1000 F (**1**) have been added to those from this investigation for material initially solution annealed at 2200 F. There is remarkably little variation in strength for reductions between 1000 and 2000 F, and that which does occur can be accounted for in terms of precipitation between 1600 and 2000 F.

The maximum rupture strengths at 1500 F were constant regardless of the hot-working temperature (Fig. 4). Again the data suggest that a recrystallization type of subgrain mechanism controls. In this case, however, it is necessary to have the amount of reduction to obtain the optimum structure decrease with increasing temperature of working. If this is not the case, there must be a complex interrelationship between cold work, recrystallization, precipitation during hot working, precipitation and agglomeration during testing, and the mechanisms of creep and rupture leading to uniformity of rupture strength.

Precipitation During Hot-Working.—The sensitivity of rupture strength at 1200 F to temperatures of reduction is related apparently to the effects of the temperature of reheating for hot working. Specifically, the low strength of material worked at 2100 F seems due to exposure to that temperature and not the effect of reduction. In other words, if the changes in the 1200 F rupture strength brought about by simply reheating to the various hot-working temperatures are subtracted out, the hot-worked rupture properties at 1200 F are primarily a function of per cent reduction and not of hot-working temperature. The suggested explanation involves some influence on the precipitation evident microscopically only after working at lower temperatures. The low strength after working at 2200 F seems due to continuous recrystallization. The drop in maximum rupture strengths for 1000 hr at 1200 F from working at 1600 to 2000 F (Fig. 34) appears related to the precipitation during hot-working. This prior precipitation also induced extensive precipitation during testing at 1200 F. Both effects would be expected to have little influence on short-time rupture strengths but should lower long-time strengths (**3**). The precipitation effects could account for the decrease in strength at 1200 F for the observed hardness after working at 1600 and 1800 F (Figs. 26 and 27). The previous work (3) has shown that hardness can increase during aging but strength decreases. The evidence, however, seems more in favor of the main influences being related to changes in structure as controlled by plastic flow of the metal.

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 788.

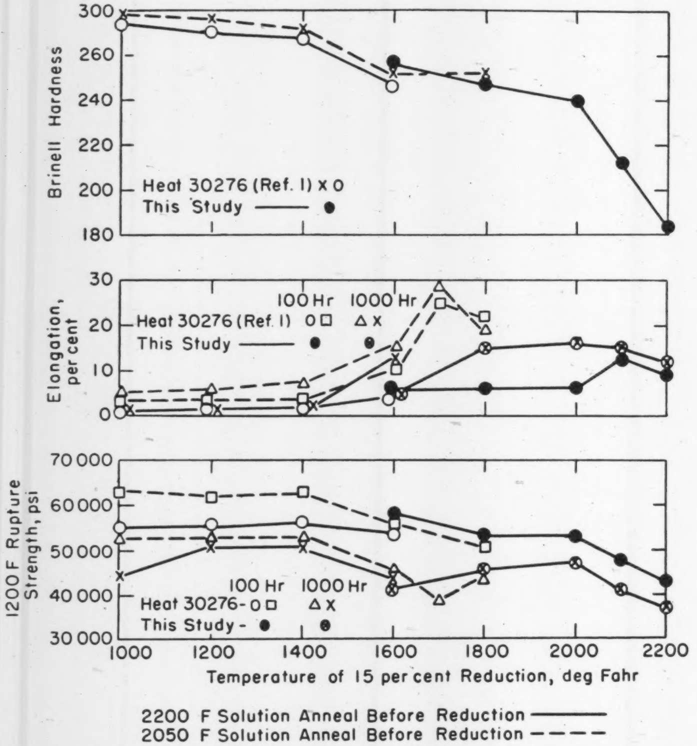

FIG. 34.—Comparison of 1200 F Rupture Strengths and Rupture Elongation, and Brinell Hardness after 15 per cent Reduction at Various Temperatures for This Investigation and Another Heat of the Same Alloy (Heat 30276, Reference 1).

Precipitation apparently had little effect on strength at 1500 F. Presumably this was due to precipitation and agglomeration during testing being so rapid and extensive that the pretesting precipitate had little influence on properties.

Ductility in Rupture Tests.—The data suggest that the same mechanisms which produce weakening in most cases lead to increased elongation and reduction of area in the rupture tests, this being particularly true for recrystallization. There are, however, aspects of the ductility relationships which do not appear to fit into this mechanism. The most difficult factor to explain is the pronounced increases in elongation at 1500 F for 100 hr resulting from simply reheating to the working temperatures (Table I) and the pronounced decreases at both 100 and 1000 hr with increasing reduction. Some influence from the precipitation reaction may be operative. The reductions for maximum strength appear to bear little, if any, relation to the reductions for minimum elongation.

Hot-Working With Decreasing Temperatures.—In working first at 2200 F and then at either 1800 or 1600 F, both the hardness and the rupture strength at 1200 F are higher than would have been anticipated from isothermal data (Figs. 26 and 27). The rupture strengths at 1200 F of material worked at 2000 and 1600 F and that given reductions of 10 per cent were also high and in accord with their hardness. Thus these working procedures developed high strength and high hardness with large total reduction. This was not quite so true for working first at 1800 and then at 1600 F. The continuously recrystallized material from working at 2200 and 2000 F had strength in agreement with its hardness.

These factors suggest an increase in the low-temperature strengthening mechanism during working without an increase in the weakening effect. The cause is not clear from the data. The alloy worked first at 2200 F may have been made more susceptible to strain hardening for a given reduction at lower temperatures. The suppression of precipitation during working at 1800 and 1600 F may have been involved. The high strengths of the material worked without recrystallization suggest that a stable structure was developed by the high-temperature working which could be given further limited reductions at lower temperatures without increasing the damage.

Cyclic Heating and Working.—Heating and working repeatedly between 1500 F and 1800, 2000, or 2200 F presented opportunities for several complicating reactions to occur. Precipitation and agglomeration were extensive when the top temperature was 1800 F, apparently the result of extensive precipitation at temperatures close to 1800 F. When the top temperature was 2000 F, precipitation at the top temperature was reduced. Presumably there was no precipitation at 2200 F.

If it is assumed that the $\frac{1}{2}$ hr at 2200 F was sufficient for nearly complete solution and recovery from prior working at 1500 F, the properties should approximate those arising from a 5 per cent reduction at 2200 F plus 5 per cent at 1500 F. Data are not available for working at 1500 F. Estimates based on available data from this investigation and Freeman, Reynolds, and Frey's survey, (1) however indicate that hardness and properties are close to those which might be anticipated on this basis and are in accord with hardness correlations of Figs. 26 through 33. The same is true for an upper temperature of 2000 F. Material worked between 1800 and 1500 F, however, had both low strength and low hardness, and the properties were low on the basis of hardness correlations in Figs. 26 through 33. The extensive precipitation and agglomeration during working at 1800 and 1500 F combined with recovery effects at 1800 F and the damage of extensive reductions at low temperatures all contributed to low strength.

Response to Heat Treatment:

The results from this investigation indicate that response to heat treatment is virtually independent of prior working conditions for heat treating temperatures in the range 2050 to 2200 F, Table V. These data are proof that the damage component from working is not permanent and can be removed by heat treatment. This still leaves the question as to the cause of variations in properties observed in practice for specific treatments. The suggestion is that they are due to unidentified heat-to-heat variations.

There are working conditions which lead to abnormal grain growth. It is recognized that for such conditions the

TABLE V.—RESPONSE TO HEAT TREATMENT, SUMMARY OF RUPTURE AND CREEP PROPERTIES AT 1200 F AND 1500 F FOR BARS ROLLED AND HEAT TREATED AS INDICATED.

Rolling Conditions	Tested at 1200 F						Tested at 1500 F					
	Rupture Strength, psi		Interpolated Rupture Elongation in 2 in., per cent		Minimum Creep Rate, per cent per hr		Rupture Strength, psi		Interpolated Rupture Elongation in 2 in., per cent		Minimum Creep Rate, per cent per hr	
	100 hr	1000 hr	100 hr	1000 hr	50 000 psi	25 000 psi	100 hr	1000 hr	100 hr	1000 hr	15 000 psi	8 000 psi
HEAT-TREATMENT AFTER ROLLING: 2050 F, 2 HR, WATER QUENCHED												
15% at 1600 F	44 500	38 000	11	11	0.028	0.00050	18 000	12 800^a	60.0	...	0.032	0.00005
15% at 1800 F	46 500	39 000^a	10	10^a	0.037	0.00040	17 500	13 500^a	50.0	...	...	0.00006
25% at 1800 F	48 000	42 000	10	20	0.040	0.00044	17 500	12 000^a	60.0	...	0.040	...
40% at 1800 F	48 500	38 000^a	15	10^a	...	...	...	...	...	...	...	...
65% at 1800 F	47 500	...	10	...	...	...	...	...	...	...	...	...
15% at 2000 F	43 000	38 000^a	...	...	...	0.00054	17 000	12 500	50.0	35.0	0.035	0.000065
25% at 2000 F	...	...	...	...	...	0.00060	17 000	12 000	50.0	35.0	0.028	...
40% at 2000 F	...	...	...	...	...	...	16 000	...	50.0	...	0.050	...
15% at 2200 F	47 000	40 500	10	8^a	0.022	0.00060	17 500	13 000^a	55.0	40.0^a	0.040	0.00012
25% at 2200 F	48 000	38 000	10	...	...	0.00064	18 500	...	50.0	...	...	...
40% at 2200 F	46 000	...	7	...	...	...	...	...	...	...	...	...
25% at 2200 F plus 15% at 1600 F	43 000	38 500^a	14	20^a	0.075	0.00053	17 000	13 000^a	44.0	...	0.042	0.00010
Rolled 10% each at 2200, 2000, 1800 and 1600 F	48 000	40 000	12	...	0.080	0.00049	18 000	13 000	50.0	40.0	0.031	...
HEAT-TREATMENT AFTER ROLLING: 2200 F, 1 HR, WATER QUENCHED PLUS 1400 F, 24 HR, AIR COOL												
25% at 1800 F	47 000	39 500	10	10	0.090	0.00035	18 000	12 500	30	12	0.014	0.00010
40% at 1800 F	47 000	40 000	12	15	0.060	0.00043	17 000	13 500^a	23	25^a	0.022	...
25% at 2000 F	47 000	41 000^a	20	10^a	0.070	...	18 500	12 500	25	15	0.020	...
40% at 2000 F	49 000	39 000	15	10	0.060	...	18 000	13 000	30	24	0.020	...
25% at 2200 F	48 000	38 000^a	11	8^a	0.085	0.00036	18 000	13 000	28	10	0.013	...
40% at 2200 F	49 000	38 000	10	14	0.085	...	18 000	13 000	30	...	0.014	...
15% at 2200 F plus 25% at 1800 F	48 000	40 000	10	15	0.050	0.00042	17 500	12 500	35	30	0.015	...

HEAT-TREATMENT AFTER ROLLING: 2050 F, 2 HR, WATER QUENCHED PLUS 15 PER CENT HOT-COLD WORKED AT 1200 F

Heat to 1800 F, ½ hr, roll 5 per cent, cool to 1500 F, roll 5 per cent, hold 2 hr, reheat to 1800 F, repeat cycle 3 more times	57 000	51 500	4	4	0.00070	0.00008	23 500	16 000	20	10	0.0010	0.00005
Heat to 2000 F, ½ hr, roll 5 per cent, cool to 1500 F, roll 5 per cent, hold 2 hr, reheat to 1800 F, repeat cycle 3 more times	55 000	49 000	4	...	0.00056	0.00004	24 000	18 000	10	7	0.0010	0.00007
Heat to 2200 F, ½ hr, roll 5 per cent, cool to 1500 F, roll 5 per cent, hold 2 hr, reheat to 1800 F, repeat cycle 3 more times	56 000	50 000	4	4	0.00080	0.000045	24 000	17 500	12	10	0.00070	0.000044

[a] **Extrapolated.**

response to heat treatment will not be independent of prior history regardless of treatment condition.

General Observations:

The relationships between hardness and properties in Figs. 26 through 33 demonstrate the reasons for the inadequacy of hardness for predicting properties at high temperatures. Large reductions at essentially constant temperature or repeated reductions with low-temperature reheats too brief to permit recovery and solution lead to low strength in relation to hardness.

No direct relationship between grain size and properties was observed. Recrystallization during working was accompanied frequently by low strength. It is doubtful, however, that grain size in itself was nearly as much a factor as strain hardening, recovery effects and possible structural alterations or precipitation effects accompanying the deformation.

The high-temperature precipitation accompanying exposure to or working in the temperature range of 1600 to 2000 F had not been observed previously. It certainly is the source of the extensive precipitates frequently observed in hot-worked products.

The reasons for or the significance of the sensitivity of the lattice parameters to working conditions and cooling rate are not understood. They do not seem explainable on the basis of ordinary solution and precipitation of odd-sized atoms and indicate that they cannot be used to estimate solubility of alloying elements as was originally intended. The lattice parameter variations were so large, however, that they pose questions as to important unidentified reactions such as ordering of the matrix structure having more effect on properties than now seems evident.

Limitation of Results:

The most serious limitation of the results appears to be the use of experimental material previously hot worked to a high degree, and then solution annealed at 2200 F to minimize this prior history. The limitations introduced by the method and conditions of working are uncertain. It is expected the general principles would remain the same even though it is difficult to foresee the effects of more rapid and larger reductions during rolling, the difference between rolling and hammer forging, influence of die constraint, etc.

The conditions of working over a falling temperature range that were investigated were extremely limited. Undoubtedly this would be a fertile field for further experimentation to cover more ranges of reductions and temperatures of reduction.

In this investigation, reasonable uniform working throughout the cross-sections was obtained. In actual practice there may be considerable variation in the metal movement within a given cross-section, which could produce variable properties across the section in the hot-worked condition. The properties at individual locations, however, should be in accordance with degree of reduction as established by this investigation.

It is believed that the general principles established apply to all alloys of the same general metallurgical type. This would include practically all of the superalloys, except those dependent on the aging reactions derived from the additions of aluminum and titanium. The amounts and temperatures of reduction for increases or decreases in strength in various alloys would be expected to vary depending on relative strain hardening and recovery characteristics during working, as well as individual structural stability during testing.

SUMMARY AND CONCLUSIONS

Principles were developed through this study which indicate the necessary controls to produce high strength and ductility at high temperatures in the hot-worked condition. Principles were established also outlining the basic causes of low strength and ductility in the hot-worked condition. It is believed that the basic principles apply to a wide range of alloys, the greatest exception being those alloys dependent upon precipitation hardening for strength. The more important principles evolved were:

1. During hot working at constant temperature only limited amounts of reduction can be introduced before further reduction either results in reduced strength or no further increase in strength at high temperatures.

For rupture strength to 1000 hr at 1200 F, the optimum reduction was 15 per cent, independent of working temperature. The maximum strength was also independent of the working temperature, except when recrystallization occurred during working or when certain previous precipitation reaction occurred during heating for working in the range of 1600 to 2000 F. Maximum rupture strength at 1500 F was constant regardless of the temperature of reduction. The reduction for optimum strength, however, varied with the temperature of reduction.

The reductions for maximum creep resistance were generally less than for maximum rupture strength.

2. Reductions over a range of falling temperatures so that the reduction at any one temperature was below that causing a fall off in strength if worked isothermally resulted in much higher strengths at 1200 F than could be obtained by isothermal reduction. Moreover the fall off in strength at 1500 F with large total reductions was avoided.

Application of this principle permits development of strengths equal to those normally obtained by hot-cold working at 1200 to 1400 F with all working temperatures in excess of 1800 F. Many small reductions over the temperature range also provide high ductility.

3. Repeated working and reheating from temperatures below 2000 F results in low strength at elevated temperatures and high ductility. Both extensive precipitation in the range of 1800 to 2000 F together with more than optimum reduction without the curative effects of high reheat temperatures appear to be involved.

It appears certain that these principles explain many of the heretofore unexplained variations in the high-temperature properties of alloys of the type considered. Furthermore, the control of hot-working conditions to produce high level properties does not seem too complex or critically dependent on working conditions.

The fundamental structural studies suggest that strain hardening is the predominant strengthening mechanism during working. Loss in strength from excessive reduction at relatively high working temperatures is associated with recrystallization during working. Apparently the same factors of large reductions which induce recrystallization at high temperatures damages the creep strength at lower temperatures where recrystallization does not occur. The indications are that this occurs first in the vicinity of the grain boundaries. Thus low-stress, high-temperature creep strength which is presumably dependent mostly on grain boundary conditions has relatively small optimum reductions. As the test stress is increased or the temperature of creep reduced so that creep becomes more a function of normal slip within the grains, larger reductions can be tolerated without damage. Apparently working over a falling temperature range avoids accumulation of damage from large reductions.

The basic mechanism was not established. Working may alter substructures. Indeed the extreme sensitivity of lattice parameter to hot working conditions and cooling rate suggests that unidentified reactions such as ordering of the matrix structure have more effect on properties than now seems evident.

The interplay of the strengthening and weakening mechanism demonstrates very clearly why such characteristics as hardness do not correlate with strength properties at elevated temperatures.

Good evidence has been presented to show that superior combinations of strength and ductility at high temperatures can be obtained by controlled hot working which have been impossible to attain by heat treatment or hot-cold working. This evidence points to control of hot working as being an extremely promising method of treatment to add to the other metallurgical manipulations commonly used to obtain improved properties at elevated temperatures.

Acknowledgment:

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EFFECTS OF CREEP STRESS HISTORY AT HIGH TEMPERATURES ON THE CREEP OF ALUMINUM ALLOYS*

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SYNOPSIS

Structural changes attending creep at high temperatures and high stresses are dependent on the preceding creep stress history. Results on aluminum solid-solution alloys indicate that initial plastic straining upon application of a constant stress decreases the creep resistance whereas the straining during creep increases the creep resistance. At low stresses the structural changes important to creep are minor. Neglecting transients that were observed upon change of stress, high-temperature creep at low stresses obeys a mechanical equation of state.

Several investigations (**1, 2, 3, 4, 5, 6**)[3] on creep of high-purity metals and dilute alpha solid solutions at high temperatures have shown that

$$\epsilon = f(\theta) \qquad [\sigma = \text{constant}] \ldots\ldots (1)$$

where:

ϵ = total plastic strain,
f = function that depends on the stress,

$$\theta = \int_0^t e^{-\Delta H/RT} dt \ldots\ldots\ldots\ldots\ldots\ldots (2)$$

ΔH = activation energy,
R = gas constant,
T = absolute temperature,
t = time under test, and
σ = applied stress.

The activation energy, ΔH, for high-temperature creep was found to be insensitive to the applied stress (**1, 2, 3**) and to the preceding creep strain and cold work (**1–6**). In fact the activation energy for creep was found equal to that for self-diffusion (**3**).

A typical correlation achieved by Eq 1 is given in Fig. 1. Since the external variables of stress and temperature were held constant throughout creep, the decreasing creep rate over the primary stage of creep must be ascribed to changes of the internal variable of structure during creep. And since the shape of the ϵ-θ curve is different for each new stress (**1**), the structural changes attending creep must be dependent on the stress. This fact is again emphasized by the data gathered in the present investigation as shown by the logarithmic plots recorded in Fig. 2.

Whereas the course of high-temperature creep at a given stress under any history of temperature in the range of rapid crystal recovery (above about one-half the absolute melting temperature) can be predicted from two alternate constant temperature creep curves with the aid of Eqs 1 and 2 (2, 3), no method for predicting creep from constant stress creep data for a variable stress history has yet been uncovered. The major difficulty in formulating such methods of prediction arises from the effect of the entire precreep stress history on the structure. The investigation described in this report was therefore initiated in an attempt to learn more about the effect of stress history on the creep of metals at high temperatures.

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 804.

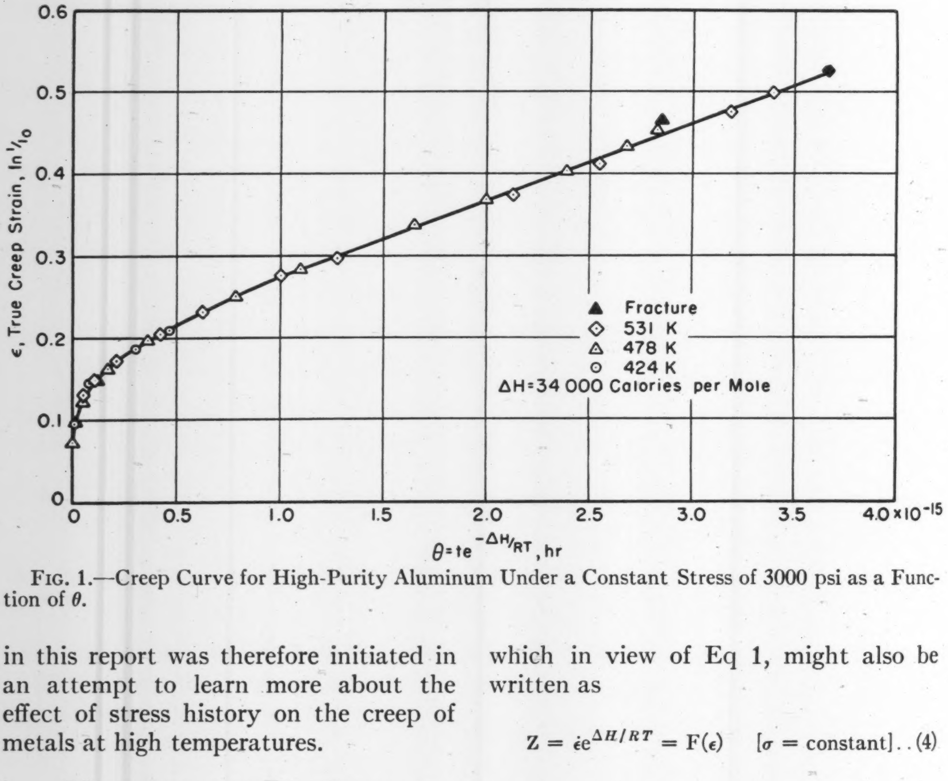


FIG. 1.—Creep Curve for High-Purity Aluminum Under a Constant Stress of 3000 psi as a Function of θ.

MATERIALS AND EQUIPMENT

The composition, annealing treatments, and grain sizes of the high-purity aluminum alloys used in this investigation are given in Table I.

All creep tests were conducted under constant stress by use of creep equipment having calibrated Andrade-Chalmers (7) type of lever arms. The following limits of accuracy were maintained:

Stress	±1 per cent
Temperature	±0.5 C
Strain	±0.0001

CREEP AT HIGH STRESSES

In order to obtain an expression for the creep rate, Eq 1 might be differentiated with respect to time, yielding

$$Z = \dot{\epsilon} e^{\Delta H/RT} = \frac{df}{d\theta} = f'(\theta) \quad [\sigma = \text{constant}] \ldots (3)$$

which in view of Eq 1, might also be written as

$$Z = \dot{\epsilon} e^{\Delta H/RT} = F(\epsilon) \quad [\sigma = \text{constant}] \ldots (4)$$

Thus the same value of Z is obtained for creep at a given stress at identical values of the strain ϵ, independent of the test temperature. Consequently, as has been proven more completely previously (2), identical structures must also be obtained following creep at a given stress to the same strain independent of the

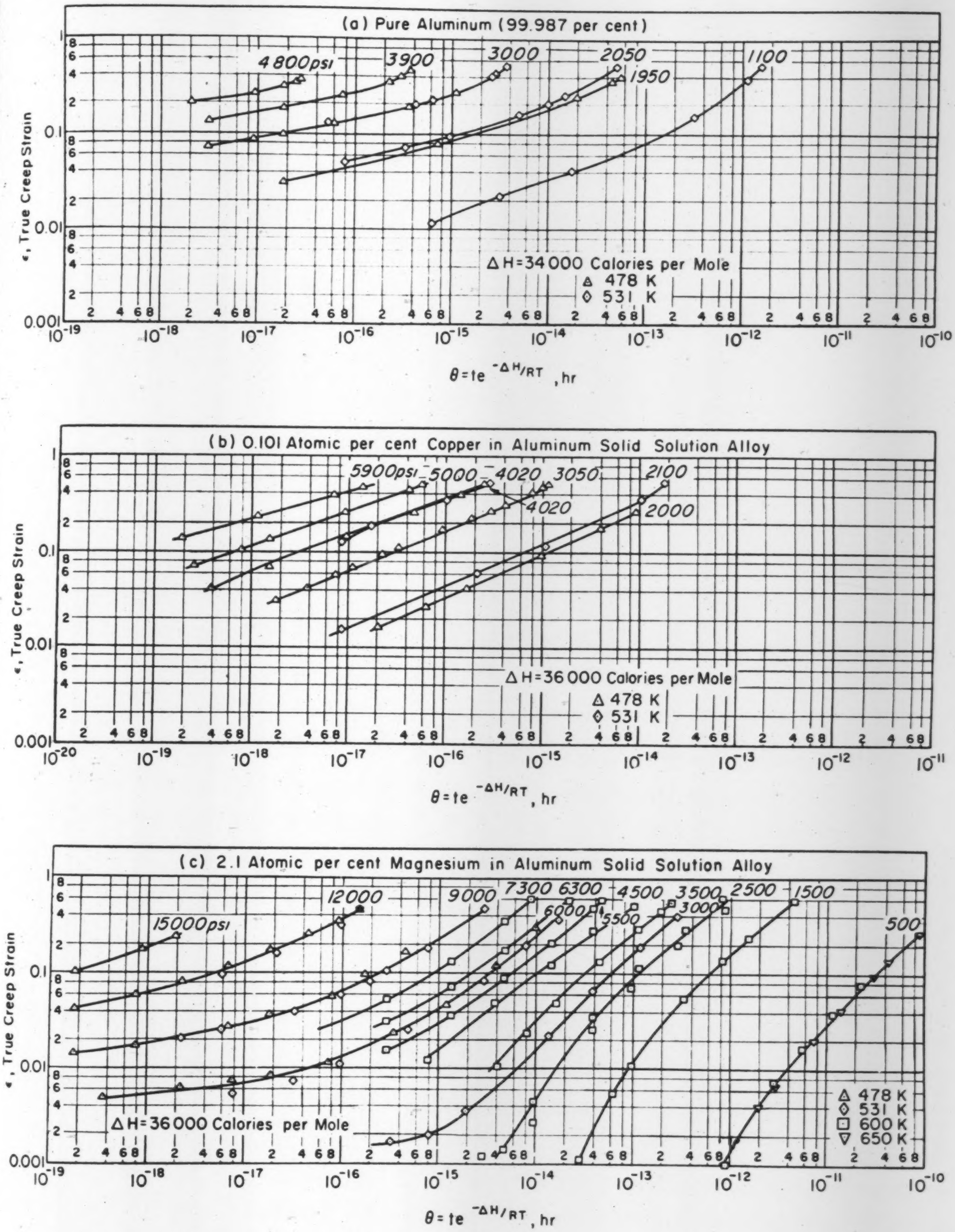


FIG. 2.—Correlation of Creep Strain *Versus* Time Data for Various Aluminum Alloys by Means of $\epsilon = f(\theta)$ Under Constant Stress.

test temperature. Typical examples of Z-ϵ correlations are shown in Figs. 3 to 5 for the three alloys investigated here.

The validity of the Z-ϵ relationship of Eq 4, as documented in the preceding paragraph, was based on the previously established validity of the ϵ-θ relationship of Eq 1. More direct experimental proof of the Z-ϵ correlations is revealed by the good coincidence of the Z-ϵ curves (Figs. 3 to 5) for a given stress at various test temperatures.

In the absence of structural changes during the course of creep, the curves for constant stress and temperature should have been horizontal lines. The nature of the observed Z-ϵ curves must therefore arise from structural changes attending creep. Furthermore the significant differences in the shapes of the Z-ϵ curves as a function of stress reveal that the structural changes attending creep are dependent on the stress history during creep. Since Z decreases over the primary stage of creep, the structure becomes more creep resistant with creep straining. And since Z decreases more rapidly with increasing values of ϵ for the highest applied stresses, the structural changes must be the greatest for the highest applied stresses.

TABLE I.—CHEMICAL COMPOSITION, ANNEALING TREATMENT, AND GRAIN SIZE OF ALUMINUM ALLOYS INVESTIGATED.

Atomic per cent, Solute	Chemical Analysis, weight per cent impurities					Annealing Treatment of Material	Grain Diameter, mm
	Copper	Iron	Silicon	Manganese	Magnesium		
Pure aluminum...........	0.006	0.003	0.003	...	0.001	30 min at 860 F (733 K)	0.21
0.10 per cent copper.......	...	0.003	0.003	0.001	0.0006	50 min at 800 F (700 K)	0.29
2.09 per cent magnesium...	0.001	0.002	0.001	0.001	...	75 min at 800 F (700 K)	0.24

The point of a minimum creep rate (or Z) corresponds to the secondary creep rate, and where Z increases with further increase in ϵ, creep has entered the tertiary stage. Inasmuch as tertiary creep might be stimulated by such factors as condensation of vacancies and significant amounts of microfracturing, it will be neglected in the following discussion as somewhat extraneous to the current problem. Major emphasis will therefore be confined only to the primary stage of creep.

According to the above arguments, stress must play two alternate roles in determining the creep rate. The first depends on the instantaneous stress and the second on the modification of structure induced by the preceding stress history. In order to isolate the first factor for independent study, a series of specimens were precrept under a given stress to the same strain whereupon the stress was decreased to a new value (8). Thus, the instantaneous creep rate following the decrease in stress pertained to the structure developed during the precreep history. In this way it was shown that for high stresses

$$Z = \dot{\epsilon}e^{\Delta H/RT} = S''e^{B\sigma} \quad \ldots\ldots\ (5)$$

TABLE II.—VALUES OF THE STRESS PARAMETER β FOR ALLOYS INVESTIGATED.

Alloy	β	Reference
Pure aluminum.....	$\frac{1}{191}$	(8)
0.1 per cent copper in aluminum alloy............	$\frac{1}{252}$	(8)
2.1 per cent magnesium in aluminum alloy.......	$\frac{1}{1860}$	This investigation, Fig. 6

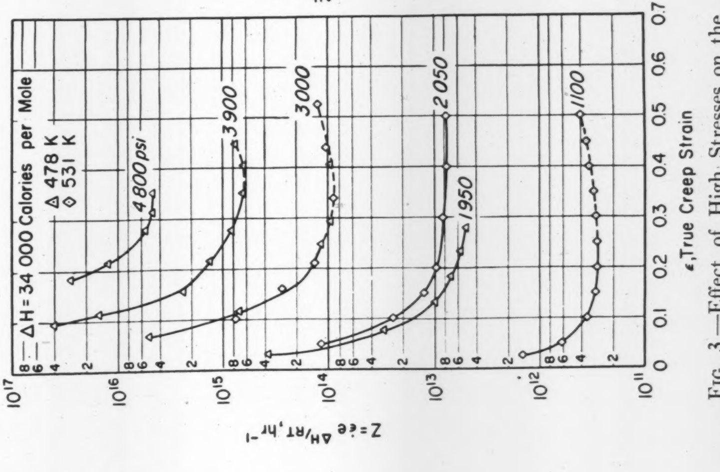


FIG. 3.—Effect of High Stresses on the Creep Rate as a Function of Strain for High-Purity Aluminum.

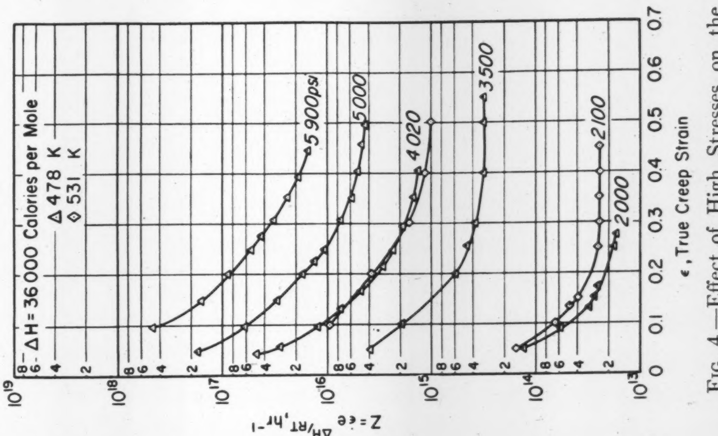


FIG. 4.—Effect of High Stresses on the Creep Rate as a Function of Strain for 0.101 Atomic per cent Copper in Aluminum Alloy.

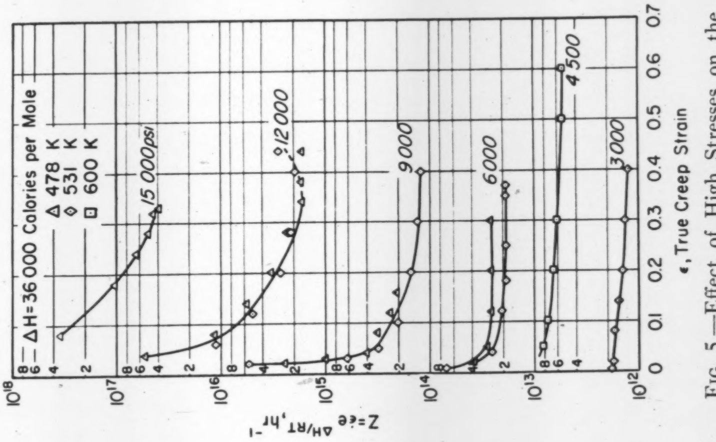


FIG. 5.—Effect of High Stresses on the Creep Rate as a Function of Strain for 2.1 Atomic per cent Magnesium in Aluminum Alloy.

where for a given alloy, B was found to be independent of temperature and the precreep history of originally annealed specimens. (Severe cold working at 78 K caused a decrease in the value of B, but following extensive creep B was restored to the value obtained for the originally annealed state) (5). Since both B and ΔH were independent of the creep strain, the structure parameter S'' was isolated as the primary factor that changes during the course of creep of initially annealed high-purity alpha solid solutions.

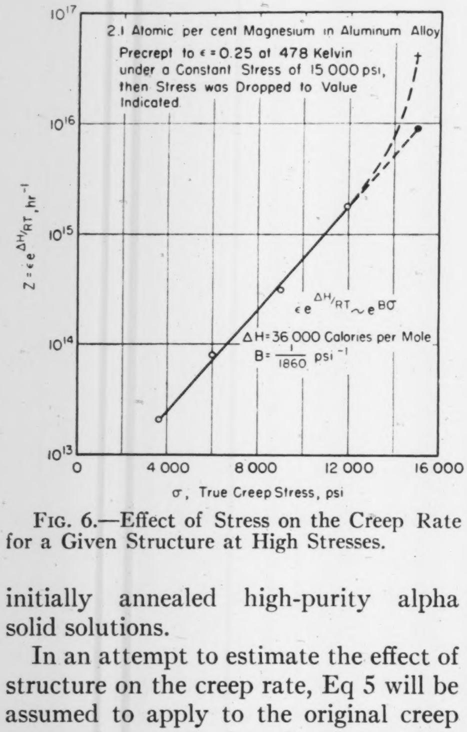


FIG. 6.—Effect of Stress on the Creep Rate for a Given Structure at High Stresses.

In an attempt to estimate the effect of structure on the creep rate, Eq 5 will be assumed to apply to the original creep curve. As proven by the decrease in stress technique, the values of B for originally annealed specimens will be taken to be constant over the entire range of strains and equal to the previously determined values recorded in Table II (8). Consequently all structural changes attending creep testing will be reflected in appropriate changes in S''. Actually, as shown in Fig. 6, a small error is introduced by the assumption that the correct creep rate–stress relationship is $\dot{\epsilon} e^{\Delta H/RT} \propto e^{\beta\sigma}$ where B is a constant. The value of Z for the original creep curve is shown by the symbol +, whereas that obtained by extrapolation of the $\dot{\epsilon} e^{\Delta H/RT} \propto e^{\beta\sigma}$ relationship is given by ●. Consequently the calculated S'' value, using the relationship, is somewhat less than the true value. The discrepancy between the true and extrapolated value increases with increasing precreep stresses. But the extrapolated value is never less than about one-fifth of the true value for the data recorded here. This factor is small in contrast to the changes of several orders of magnitude in S'' encountered during the course of creep. The values of the structure parameter, S'', obtained in the above-mentioned way, are recorded as a function of the total strain in Figs. 7 to 9. The data for high-purity aluminum in Fig. 7 suggest that two factors affect the S'' values. During the course of creep S'' decreases, the decrease being greatest for the highest stresses. But the intersection of several of the curves for various stresses suggests that S'' increases abruptly during initial straining immediately upon application of the stress. At strains preceding the intersection of two S''-ϵ curves, the higher stress induces the lesser creep-resistant structure, but beyond the point of intersection the higher stress induces the greater creep-resistant structure. Such intersections of the S''-ϵ curves for various stresses are not noticed in the data of Fig. 8 for the 0.101 atomic per cent copper alloy. The trends here are similar to those for the high-purity aluminum at strains beyond the points of intersection. Thus, the most creep-resistant structures are those obtained following creep at the highest stresses.

At first glance, the data for the 2.1 atomic per cent magnesium alloy, given

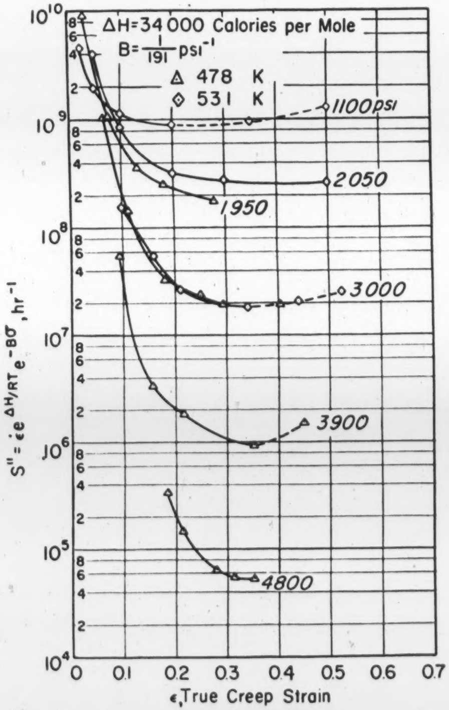


FIG. 7.—Effect of High Stresses on the Structure Parameter-True Creep Strain Relationship for High Purity Aluminum.

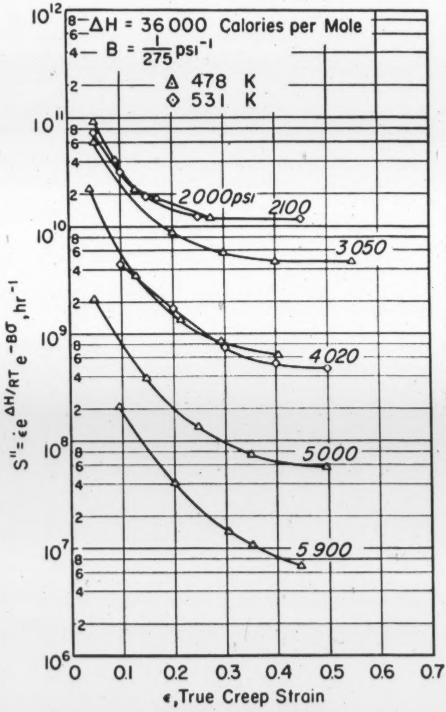


FIG. 8.—Effect of High Stresses on the Structure Parameter-True Creep Strain Relationship for 0.101 Atomic Per Cent Copper in Aluminum Alloy.

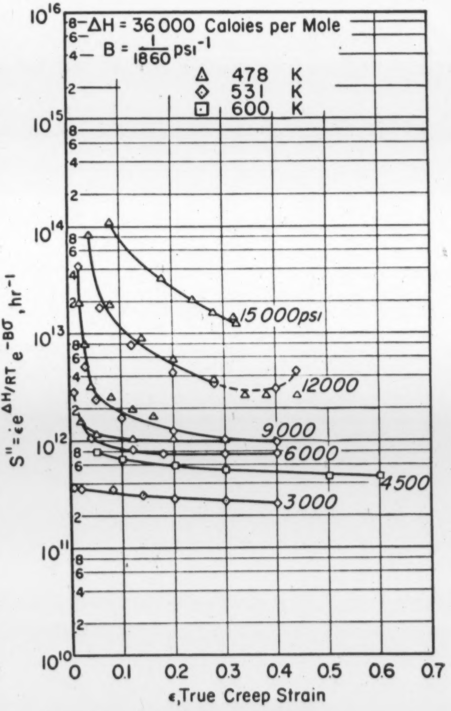


FIG. 9.—Effect of High Stresses on the Structure Parameter-True Creep Strain Relationship for 2.1 Atomic Per Cent Magnesium in Aluminum Alloy.

in Fig. 9, appear to be contradictory to the data for the remaining alloys insofar as the highest stress yields the highest values of S'' and thus gives the least creep-resistant structure. This apparent anomaly, however, might be rationalized by the following observations: First, the initial straining upon application of the stress appears to cause a greater increase in S'' for the 2.1 atomic per cent magnesium alloy than for the high-purity aluminum. And secondly, the decrease in S'' with creep straining is much less than that observed for high-purity aluminum. Consequently S'' is found to be greater for high stresses over the entire range of strains that were investigated for the 2.1 atomic per cent magnesium alloy.

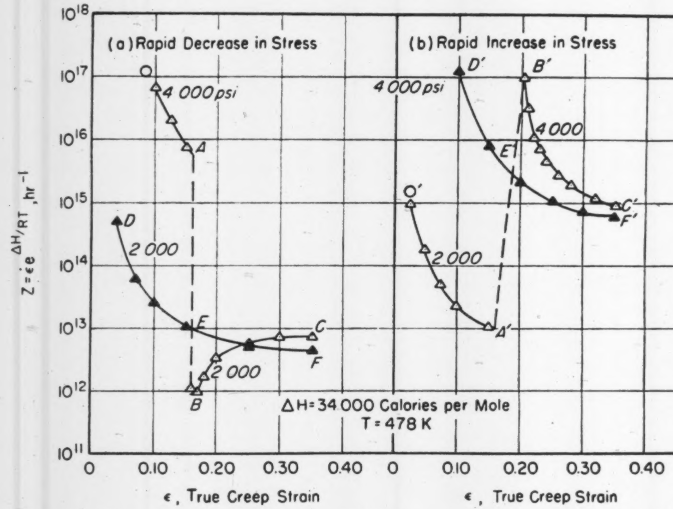


FIG. 10.—Effect of Change in Stress on Subsequent Rate of Creep for High-Purity Aluminum.

The rationalization of the S''-ϵ data given in the preceding paragraph suggests that the creep behavior of the various alpha solid solutions of aluminum are qualitatively similar: Initial straining results in an increase in S'' and therefore introduces a less creep-resistant structure; creep straining over the primary stage causes S'' to decrease and thus introduces a more creep-resistant structure. Quantitatively, however, there are significant differences: The increase in S'' for the 2.1 atomic per cent magnesium alloy upon initial straining is much greater than that for high-purity aluminum; and the decrease in S'' for the 2.1 atomic per cent magnesium alloy with creep straining is much less than that for the high-purity aluminum. The remaining aluminum-copper alloy appears to fall into an intermediate category between those for high-purity aluminum and the 2.1 atomic per cent magnesium alloy.

Special experiments recorded in Figs. 10 and 11 were conducted to provide additional checks on the above-quoted deductions on the effect of initial straining and creep straining on the structure-sensitive parameter S''. As shown in Fig. 10 the Z-ϵ curve for pure aluminum under 2000 psi is given by *DEF*; the curve *OABC* was obtained by creep straining to 16 per cent total strain at 4000 psi and then reducing the stress to 2000 psi. Inasmuch as no pronounced transients were observed upon decrease in the stress, the structure at *A* and *B* must be presumed to be the same. And since Z_B is less than Z_E while the stresses are identical at *B* and *E*, precreep of pure aluminum to 16 per cent strain at 4000 psi provides a more creep-resistant structure than precreep to the same strain at 2000 psi. The Z-ϵ curve *D′E′F′* was that obtained upon stressing exclusively at 4000 psi, whereas curve *O′A′B′C′* was obtained by precreeping to 16 per cent total strain at 2000 psi and then abruptly changing the stress to 4000 psi. If the change in Z upon going from *A′* to *B′* were exclusively due to the imposed change in stress, log $Z_{B'}$ − log $Z_{A'}$ should have been equal to log Z_A − log Z_B; but log $Z_{B'}$ − log $Z_{A'}$ was slightly greater than log Z_A − log Z_B despite the identity of the stress changes involved. Consequently, in harmony with the deductions based on the intersecting S''-ϵ curves of Fig. 7, it must be concluded that the instantaneous strain obtained upon increasing the stress from 2000 to 4000 psi introduces a slightly less creep-resistant structure.

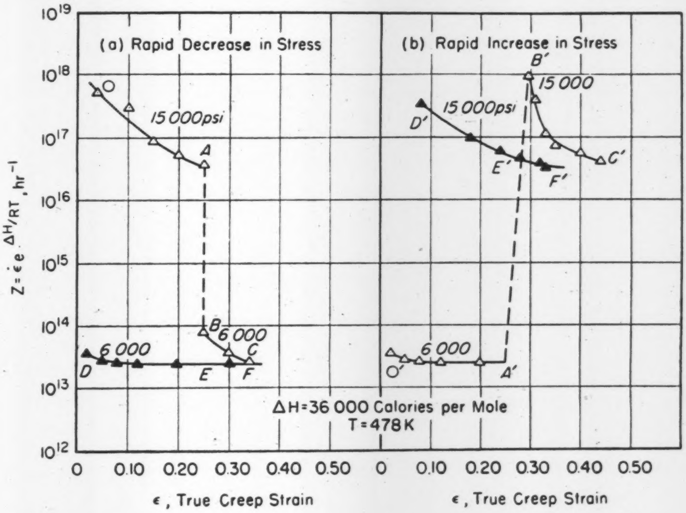


FIG. 11.—Effect of Change in Stress on Subsequent Rate of Creep for 2.1 Atomic per cent Magnesium in Aluminum Alloy.

More conclusive evidence for this explanation is given by the data recorded in Fig. 11 for the 2.1 atomic per cent magnesium alloy. From the S'' *versus* ϵ curves of Fig. 9 it was suggested that the increase in S'' upon initial straining of the 2.1 atomic per cent magnesium was much greater than that for the high-purity aluminum. Consequently, it might be presumed that this effect is also great upon increasing the stress following a precreep history at some lower stress. The Z-ϵ curve *DEF* was obtained by creeping exclusively at 6000 psi, whereas the curve *OABC* was obtained by creeping 25 per cent at 15,000 psi and then abruptly decreasing the stress to 6000 psi. Since Z_B is above Z_E in spite of the identity of stress, the precreeping at 15,000 psi leads to a less creep-resistant structure than precreeping at 6000 psi. Again the difference log Z_A − log Z_B is attributable exclusively to the change in stress. Curve *D′E′F′* was obtained upon creep exclusively at 15,000 psi, whereas curve *O′A′B′C′* was obtained by precreeping to 25 per cent

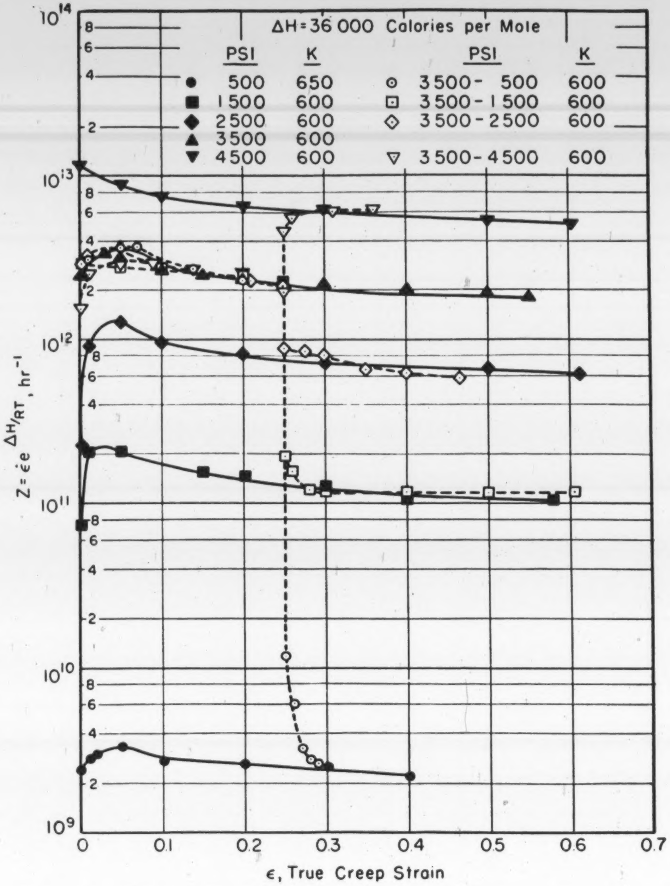


FIG. 12.—Effect of Low Stresses on the Creep Rate as a Function of Strain for 2.1 Atomic per cent Magnesium in Aluminum Alloy.

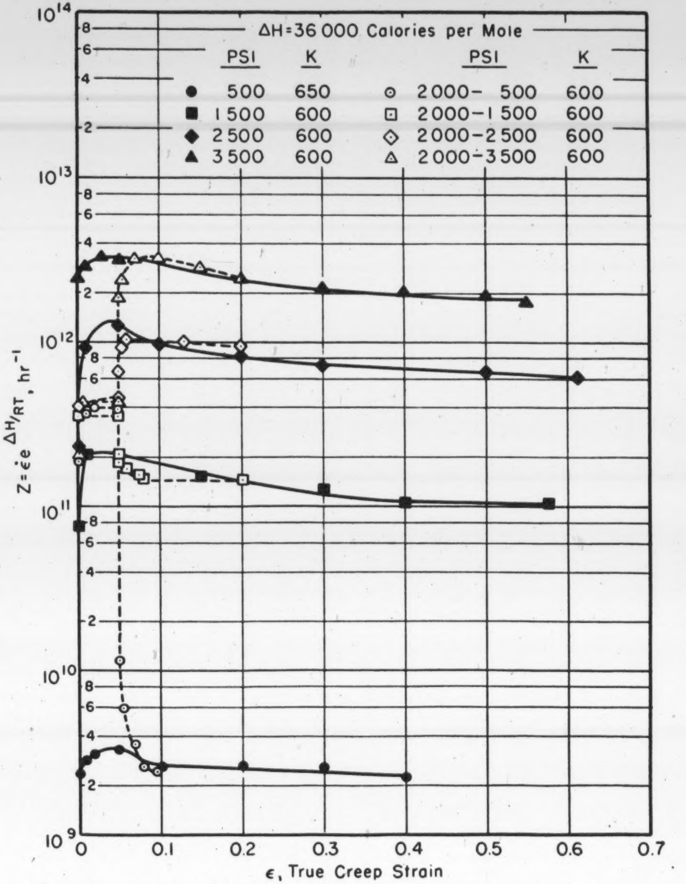


FIG. 13.—Effect of Low Stresses on the Creep Rate as a Function of Strain for 2.1 Atomic per cent Magnesium in Aluminum Alloy.

at 6000 psi and then changing the stress abruptly to 15,000 psi. If no change in structure had occurred upon increasing the stress, log $Z_{B'}$ — log $Z_{A'}$ should have equalled log Z_A — log Z_B; since log $Z_{B'}$ — log $Z_{A'}$ is much greater than log Z_B — log Z_A it is necessary to conclude that the instantaneous straining upon increase in the stress led to a much less creep-resistant structure.

Two causes for the failure of a mechanical equation of state for creep at high temperatures and high stresses are now apparent: the structure changes upon initial creep straining in such a way as to produce a less creep-resistant material. And during creep a more creep-resistant structure is produced. Thus dissimilar structures are obtained by different stress histories. After extensive creep at a new stress, however, as shown by the data of Figs. 10 and 11, the structure approaches a steady-state value that is dictated by the new stress.

CREEP UNDER LOW STRESSES

Two factors suggest the creep law for metals might undertake a more simplified form at sufficiently low stresses. As the applied stress is reduced, the initial plastic straining becomes less and finally vanishes at some yet finite value of the stress. In this range, then, the initial structure immediately following stressing should be that for the annealed state. Furthermore, as shown in Figs. 3 to 5, the change in structure upon continued creep straining is less for creep at lower values of the stress. Under appropriate limiting conditions, it is possible that the structure-sensitive parameters of the creep laws are substantially constant throughout the course of creep. Under such limiting conditions creep should obey a mechanical equation of state.

Previous investigations (9) have shown that for such low stresses where the initial plastic strain is zero, the initial creep rate is correlated with the stress by the relationship

$$Z = \dot{\epsilon}e^{\Delta H/RT} = S'\sigma^n \ldots\ldots\ldots \quad (6)$$

If a mechanical equation of state were valid, S' and n would have to be constant independent of any preceding creep history.

In order to check the validity of these precepts, the tests recorded in Figs. 12 and 13 were undertaken. These data reveal that the creep curve at constant low stresses (shown by the solid lines) are somewhat dissimilar from the curves obtained at higher stresses. In all of the cases reported here, the initial plastic strain obtained during stressing was zero. Immediately upon stressing, a small finite creep rate was obtained, the magnitude of which increased with increasing stress. But thereafter, instead of exhibiting the usual primary stage of decreasing creep rates, the creep rate increased, rapidly acquired a maximum value, and then decreased quite slowly as creep continued, somewhat suggestive of the usual primary stage of creep. The rate of decrease of creep rate with strain was less for the lower stresses and, for the range of stresses investigated here, never exceeded a factor of one-half. As shown in Fig. 14, neglecting the initial transient phase of creep,

$$\dot{\epsilon}e^{\Delta H/RT} \simeq S'\sigma^n$$

where S' and n are practically independent of the creep strain. Thus, as shown in Fig. 15, S' is practically constant over the entire range of strains for creep at low stresses if the initial transient creep is excluded from the correlation. Within these limitations, therefore, creep at low stresses obeys a mechanical equation of state.

In order to uncover additional information on the phenomenon of creep at low stresses, an abrupt change in stress was introduced as shown by the broken

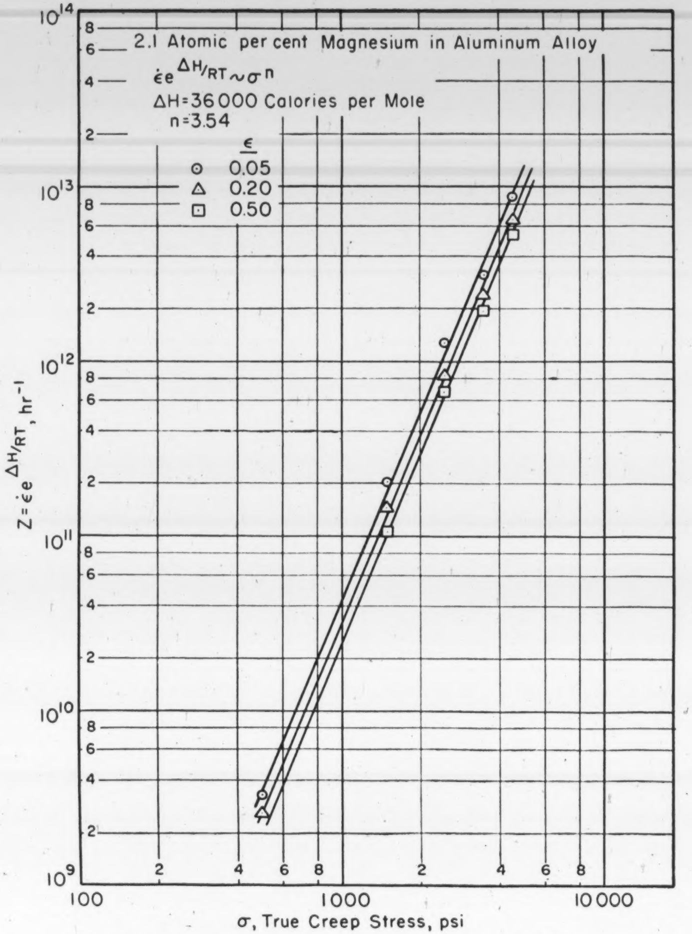


FIG. 14.—Effect of Stress on the Creep Rate at Low Stresses.

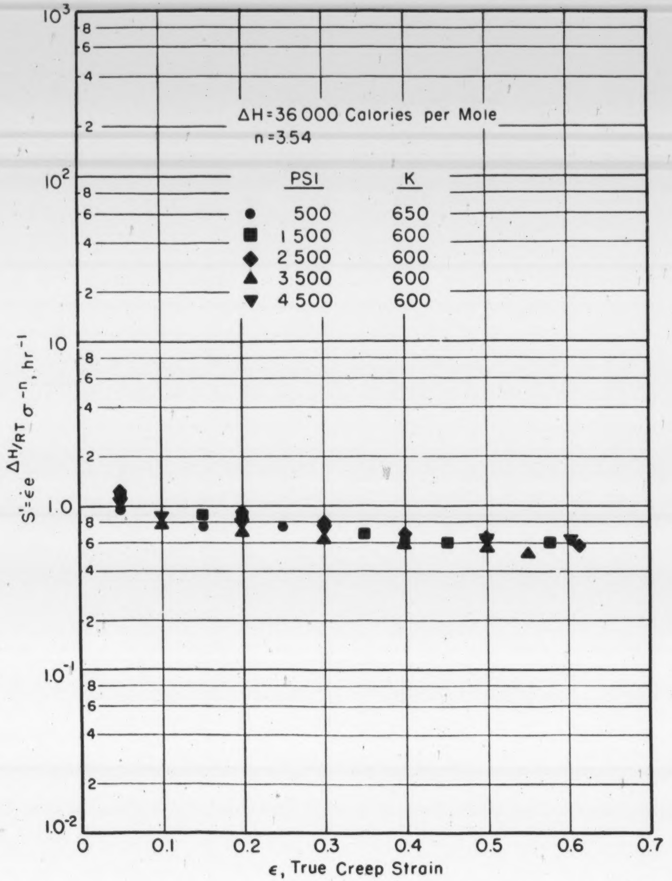
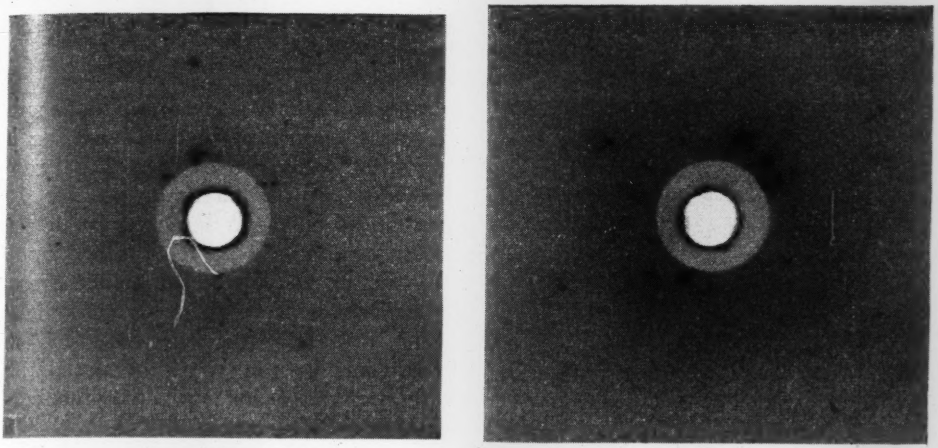
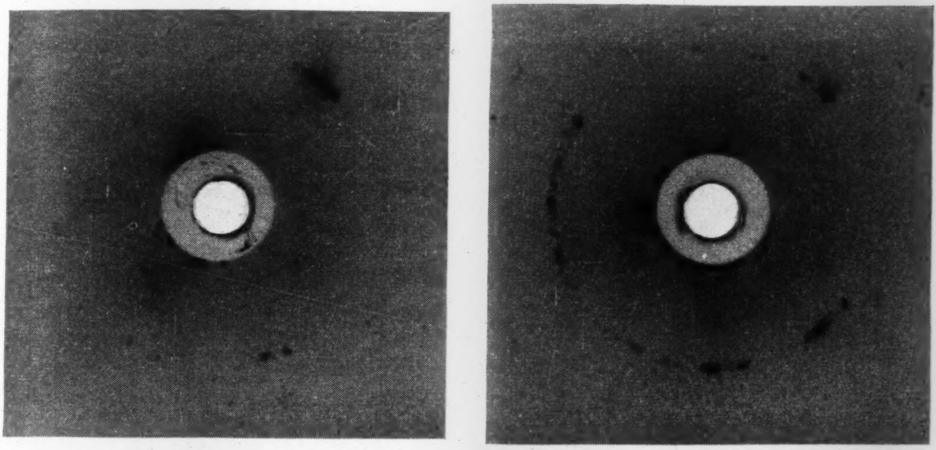


FIG. 15.—Effect of Low Stresses on the Structure Parameter-True Creep Strain Relationship for 2.1 Atomic per cent Magnesium in Aluminum Alloy.



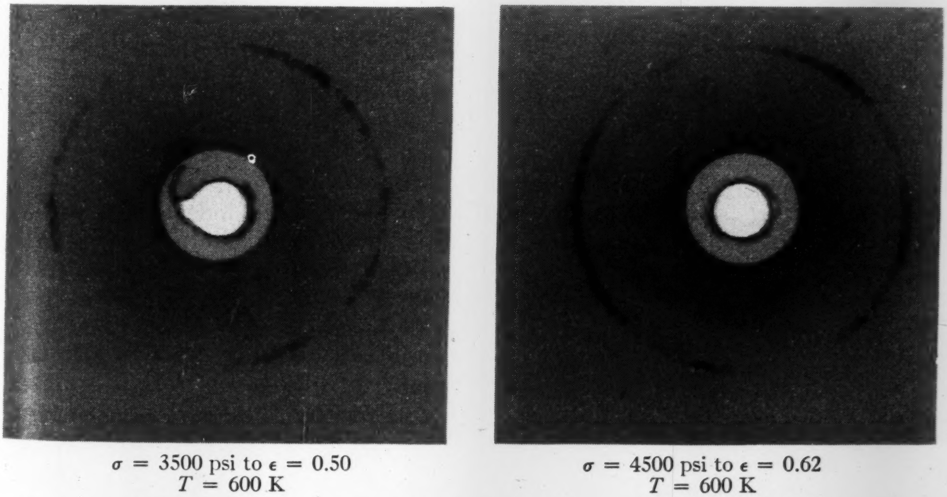
Annealed material

σ = 500 psi to ϵ = 0.40
T = 650 K



σ = 1500 psi to ϵ = 0.58
T = 600 K

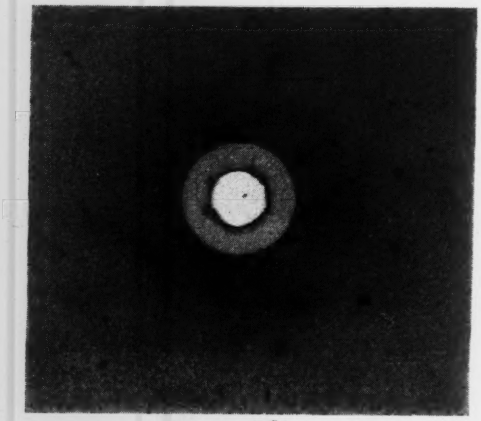
σ = 2500 psi to ϵ = 0.61
T = 600 K



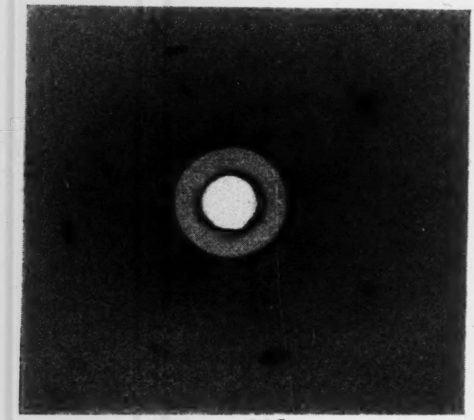
σ = 3500 psi to ϵ = 0.50
T = 600 K

σ = 4500 psi to ϵ = 0.62
T = 600 K

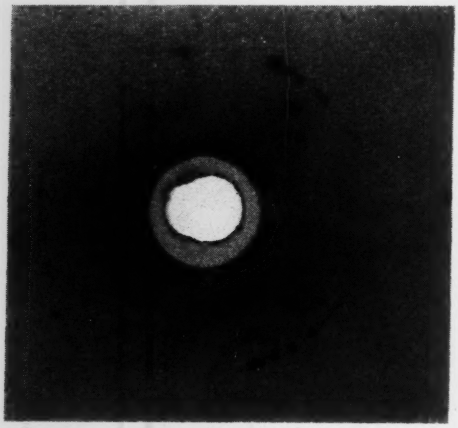
FIG. 16.—X-Ray Photograms of 2.1 Atomic per cent Magnesium in Aluminum Alloy Deformed in Creep at Various Constant Stresses.



$\epsilon = 0.01$



$\epsilon = 0.05$



$\epsilon = 0.10$

FIG. 17.—X-Ray Photograms of 2.1 Atomic per cent Magnesium in Aluminum Alloy Deformed in Creep at a Constant Stress of 3500 psi to Various Strains. (T = 600 Kelvin).

curves of Figs. 12 and 13. Such changes in stress again introduced transients: if the stress were increased the initial rate was less than the maximum rate for the new stress but soon acquired about the same steady-state value obtained upon creeping exclusively at the higher stress; if the stress were reduced, the initial creep rate following the reduction in stress was greater than the maximum rate appropriate for creeping exclusively at the reduced stress, but following a short interval the creep rate finally reduced to that which was obtained by creep exclusively at the lower stresses. These observations reveal that Eq 6 accurately represents the steady-state creep phenomenon independent of previous low-stress history; but a brief transient condition is introduced at each change of stress. Occasionally the creep rates during such transients differ by a factor of five from the steady-state values. And since the transients often persist for strains as large as 2 per cent, unqualified application of the mechanical equation of state to creep at low stresses is not completely justified.

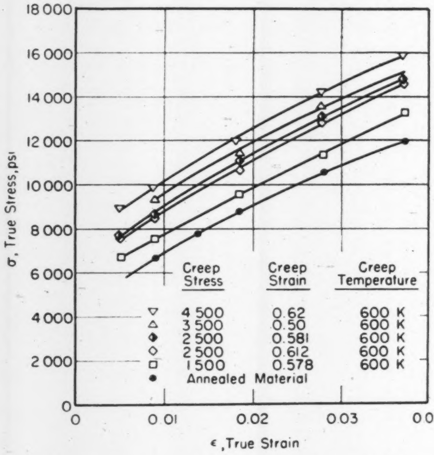


FIG. 18.—Atmospheric Tensile True Stress-True Strain Curves of 2.1 Atomic per cent Magnesium in Aluminum Alloy After Creep at Various Stresses.

CREEP AND SUBGRAIN STRUCTURE

Extensive investigations have been conducted by Wood and his colleagues (10) as well as others (2, 11, 12) on the changes in subgrain structure with creep. These investigations have shown that the original grains exhibit deformation banding in the initial phases of creep and that finally small polygonized subgrains are produced. The authors of this report have previously shown (2) that the observable substructure is a function of $te^{-\left(\frac{\Delta H}{RT}\right)}$ for a given stress in harmony with the creep strain relationship given by Eq 1. Thus the same substructure was obtained at the same values of strain independent of the test temperature for creep at a given stress. Higher stresses caused greater deformation banding in the early stages of creep and resulted in finer polygonized subgrains during the secondary stage of creep. These observations of the parallelism between the structure and creep properties suggested at one time that the changes in creep properties during primary creep was a result of these observable structural changes. But subsequently it was found that cold work improved the creep resistance although in this case less complete polygonization was observed (13). Thus it was deduced that if the structures revealed by fine pinhole back-reflection Debye-Scherrer X-ray patterns had an influence on the creep properties, other yet unrevealed factors must also play an important role. Such hidden factors might well have been specific patterns of dislocations.

Additional attempts were made in the present investigation to clarify the issues surrounding the correlation of structure as revealed by Debye-Scherrer photograms with properties. As shown in Fig. 16, the structures produced at higher stresses within the low-stress range result in greater polygonization and disorientation of the polygonized subgrains than those produced at about the same strains for the lower stresses, in spite of the identity of the structure-sensitive parameter S' for creep. Furthermore, as shown by the Debye-Scherrer back-reflection photograms of Fig. 17, a substantial change takes place in the structure during creep at one stress during which the creep rate and S' are substantially constant. Both of these observations refute any correlation between the degree of polygonization and the extent of disorientation among the polygonized subgrains and the high-temperature creep characteristics of metals. As shown by the data of Fig. 18, however, the extent of polygonization during creep is correlatable with the atmospheric temperature tensile properties.

CONCLUSIONS

1. Structural changes during initial plastic straining upon application of a stress decrease the creep resistance. This effect is small for high-purity aluminum but was quite pronounced for an aluminum alpha solid solution containing 2.1 atomic per cent magnesium.

2. Changes attending primary creep lead to higher creep-resistant structures. These effects increase with increasing stress and they are greater for high-purity aluminum than for an alpha solid-solution aluminum alloy containing 2.1 atomic per cent magnesium.

3. The structural changes attending creep at high stresses greatly influence the creep resistance of metals. At low stresses, however, the structural changes important to creep resistance are minor. To a first approximation, high-temperature creep at low stresses obeys a mechanical equation of state. Complete validity of a mechanical equation of state, however, is not achieved due to transients which result whenever the stress is changed.

4. The polygonization attending creep, as revealed by Debye-Scherrer X-ray back-reflection photograms does not correlate with the creep properties. They do, however, correlate with atmospheric temperature tensile properties.

Acknowledgments:

This investigation was sponsored by the Office of Naval Research. The authors wish to thank the O.N.R. staff for their interest and full cooperation during this study as well as related studies on the plastic properties of metals. The alloys used, together with their chemical analyses, were graciously supplied by the Alcoa Research Laboratories, Aluminum Company of America. The authors wish to acknowledge the assistance of A. Goldberg and J. Lytton who contributed materially to this investigation. To Mrs. G. Pelatowski sincere appreciation is extended for her preparation of the figures for this report.

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DISCUSSION

MR. RAY W. GUARD[1] (*by letter*).—The results presented by the authors show again the complexity of the creep behavior of relatively simple materials. The creep behavior at large strains ($\epsilon > 0.05$) is now well documented and for this test region only one comment seems pertinent. Microexamination of many creep specimens of high-purity aluminum (99.995 per cent aluminum) failed to show any voids or micro-cracks even after the creep was well into the "tertiary" stage. There remains the possibility for this material that the increase in rate is the result of some structural change which has its origin much earlier in the creep test. This hypothesis would suggest that the stress- and temperature-history effects arise because the instantaneous stress, strain, and temperature determine the rate of change of properties rather than the properties themselves.

The observations of transients in low stress tests where a change in stress is made is interesting. Their presence would seem to contradict the authors' conclusion that structural changes are of minor importance at low stresses. On the contrary, the data show that creep at a particular stress gives a structure which is less resistant to creep at lower stresses and more resistant to creep at higher stresses than specimens run at constant stress. If one uses the data of the authors' Fig. 12 to calculate the stress dependence using the method of reference (**8**),[2] a relation of the type of Eq 6 is found except that n is 2.65 rather than 3.54 as found using initial rates. In view of the early transient behavior this would seem to be a more correct procedure.

The conclusions on the relation between creep and subgrain structure suffer from the insensitivity of the X-ray method used. While the grosser aspects of the "polygonized" structure may not correlate with creep behavior, it remains plausible that fine details of structure are still important in determining the creep behavior. This hypothesis also seems to be in agreement with the previous comments in that:

1. The fine details would be expected to be much more sensitive to test conditions. Changes in fine structure could then account for the observed transient behavior on stress changes.
2. The presence of a real increase in creep rate ("tertiary" creep) can be related to changes in fine details within a polygonized structure.
3. The observed rate increase initially in low stress tests is more likely to be connected with events on a small scale than with gross features of the structure.

One aspect of the deformation behavior which was not mentioned is the effect of boundary migration. Did the authors examine specimens internally metallographically after creep for evidence of such boundary motion (that is, increase in grain size, crooked boundaries, etc.)?

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[1] Alloy Studies Research, General Electric Co., Research Laboratory, Schenectady, N. Y.

[2] The boldface numbers in parentheses refer to the list of references appended to the paper, see p. 804.

MESSRS. O. D. SHERBY, T. A. TROZERA, AND J. E. DORN (*authors' closure*).—The authors sincerely appreciate the documentation of this paper by the supplementary comments and discussions by Mr. Guard. They are tempted to agree with Mr. Guard that considerable weight must be given to his suggestion that the initiation of tertiary creep might have its origin as a result of processes that occur throughout the primary and secondary stages of creep. Up to the present, however, no analysis can be given as to how the contribution of the tertiary process to the observed primary and secondary creep rates might be estimated. In any event, it appears that Eq. 1 is reasonably valid over the entire range of high-temperature creep curves, suggesting that the tertiary process is dependent on the previous creep history. Mr. Guard's negative evidence expressed in his failure to observe voids or microcracks, however, cannot be extrapolated to a generalization regarding the active mechanism for the initiation of tertiary creep.

Using the same prima facia experimental evidence, Mr. Guard arrives at a distinctly different interpretation of the data from that presented by the authors. Mr. Guard carefully pointed out that the value of n of Eq. 6 deduced from the data of Fig. 12 using the instantaneous creep rates just before and just following a change in stress is 2.65. He contends that this value should have been used in documenting Fig. 15, whereupon, by this analysis, S' would have had higher values for the higher stresses, proving that a mechanical equation of state is not even approximated for creep at low stresses. In contrast, the authors contend that the instantaneous rates following a change in stress are representative of a transient condition in these low stress tests and therefore the steady state values should be employed in these cases. The basis of the authors contention lies in the observations recorded in reference (9) that n is about equal to 3.54 when the initial creep rates immediately following stressing are employed, coupled with the fact that n is also 3.54 when the steady state values are used as shown by the correlations given in Fig. 15 of this paper. Obviously the structures are the same immediately upon stressing in the low stress range since the initial strain is zero. Since the same value of $n = 3.54$ also applies to the steady state condition regardless of previous strain history, the structure must not have been modified. This then suggested that the differences between the initial creep rate upon increase or decrease of stress and the subsequent steady state creep rate was due to some transient phenomenon, and not to a fundamental structural change. Upon careful review of the facts the authors continue to believe that the evidence favors their interpretation of these data.

The authors agree with Mr. Guard's suggestion that the fine substructure may prove to be a key to the understanding of the effects of structural changes attending creep on the creep rate. Unfortunately no unambiguous investigations have thus far been made on this subject. No metallographic studies were made in this investigation on grain boundary shearing or grain boundary migration.

FACTORS AFFECTING THE FORMING PROPERTIES OF SEVERAL COPPER ALLOYS IN STRIP FORM*

By John T. Richards and Ellsworth M. Smith[1]

Synopsis

Bend, tension, and hardness tests were performed on beryllium copper, phosphor bronze, brass, nickel-silver, and gilding strip to determine relative forming characteristics. Formability is expressed as the minimum safe radius for cold forming a 90-deg bend. The effects of composition, temper, thickness, grain size, and grain direction are considered. This investigation supplements earlier work on the forming properties of spring materials.

In the fabrication of flat springs, contacts, clips, and small structural members from strip, bending properties provide a convenient means of evaluating formability. Safe forming radii can be determined by cold-forming strip specimens to a 90-deg bend with suitable tooling. This test procedure is not only rapid but also provides a direct measure of forming characteristics.

Bend tests on copper products utilizing a punch-and-die arrangement have been described elsewhere (**1, 2, 3**).[2] The results obtained have proven extremely valuable in problems of design and temper selection. In the present investigation, the test procedure has been modified so that specimens were bent around a radius block by means of a forming head. Experience indicates that the values obtained from both methods provide useful data relating to bending resulting from punch press or multislide work. Gohn (**4**) has also recently shown that brake-forming characteristics compare favorably with those observed in a punch-and-die setup.

As a means of expanding these earlier investigations, considerable attention has been directed toward controlling raw material variables through the use of selected lots of strip. Consequently, the material factor becomes constant so that other variables may be critically examined. The effects of temper, strip thickness, grain direction, and grain size upon bending and tensile properties for given chemical compositions are presented. This study represents a continuation of an earlier one limited to beryllium copper (**3**).

Materials

The materials investigated included: beryllium copper (alloys 25, 165, and 10), phosphor bronze (grades A, C and D), nickel silver (12 and 18 per cent spring alloys), gilding (95-5) and brass (70-30).

In order to hold the composition constant, a single coil of base stock 0.090 in.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] Chief Engineer, and Chief Metallurgist, respectively, Penn Precision Products, Inc., Reading, Pa.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 822.

thick was selected for each alloy. Lots A and B were then processed from these coils. In the case of lot A, strip was cold rolled to 0.050 in. thick, annealed or solution treated, then further rolled to produce tempers ranging from quarter hard to extra spring. Lot B was rolled from 0.090 in. to 0.032 in. and then strand annealed to produce grain sizes ranging from 0.005 mm to 0.070 mm. These coils of varying grain size were then further reduced to give the required tempers. Lot C represents material taken from stock at random in order to provide comparative data on other lots and thicknesses.

TABLE I.—CHEMICAL COMPOSITION.

	Composition, per cent									
	Beryllium Copper			Phosphor Bronze			Nickel-Silver		Brass	
	25	165	10	A	C	D	12 per cent	18 per cent	95-5	70-30
Beryllium	1.92	1.72	0.47	...	...	...	...	...	...	...
Cobalt	0.28	0.28	2.45	...	...	...	...	...	...	...
Iron	0.10	0.17	0.06	trace	trace	trace	0.10	0.09	0.008	0.015
Silicon	0.09	0.12	0.04	...	...	...	...	...	...	...
Aluminum	0.07	0.10	0.05	...	...	...	...	...	...	...
Nickel	0.01	0.03	0.01	...	...	...	12.30	17.70	...	...
Tin	trace	0.02	0.00	4.45	8.36	9.98	...	...	...	0.00
Lead	0.00	0.00	0.00	trace	trace	trace	trace	trace	trace	0.008
Manganese	...	...	...	...	...	...	0.22	0.23	...	...
Phosphorus	...	...	...	0.17	0.08	0.08	...	...	...	...
Zinc	0.00	0.01	0.01	trace	trace	trace	30.12	27.76	balance	balance
Copper	balance	balance	balance	95.36	91.54	89.92	57.24	54.20	95.42	70.60

The composition of the base stock used for each alloy in producing lots A and B is given in Table I, while temper designations and cold rolling schedules will be found in Table II.

TEST METHODS

Tension, hardness, and cold-bend tests were employed. Average grain sizes were estimated by the comparison method. Tension tests were conducted on a 10,000-lb Olsen testing machine.

The 90-deg bend test utilized a model I Di-Acro Bender. Sheared specimens ½ by 2 in. were removed from test strip 0, 45, and 90 deg to the direction of rolling. Specimens were then bent around radius blocks and pins ranging from sharp (0 radius) up to approximately ¼ in. Preliminary tests indicated good correlation with the punch-and-die setup previously employed (3).

In this study, two end points were

TABLE II.—TEMPER DESIGNATIONS.

Designation[a]	Temper	Rolled B&S Numbers Hard	Cold-Rolled Reduction in Thickness, per cent
A	Soft	0	0
¼ H	Quarter Hard	1	11
½ H	Half Hard	2	21
¾ H	Three Quarter Hard	3	29
H	Hard	4	37
XH	Extra Hard	6	50
S	Spring	8	61
XS	Extra Spring	10	69

[a] Suffix "T" indicates rolled to temper shown and age hardened 3 hr at 900 F.

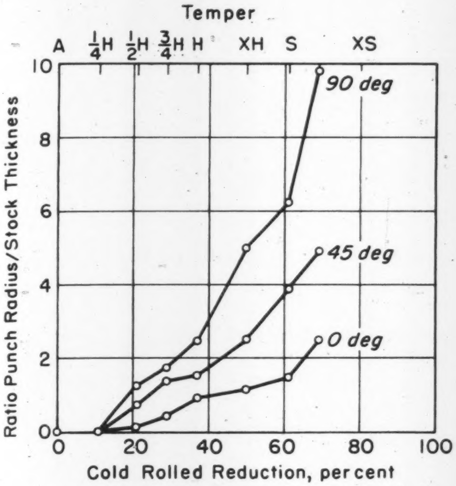

FIG. 1.—Relative Formability of Phosphor Bronze Grade D Strip with Respect to Grain Direction. Lot A.

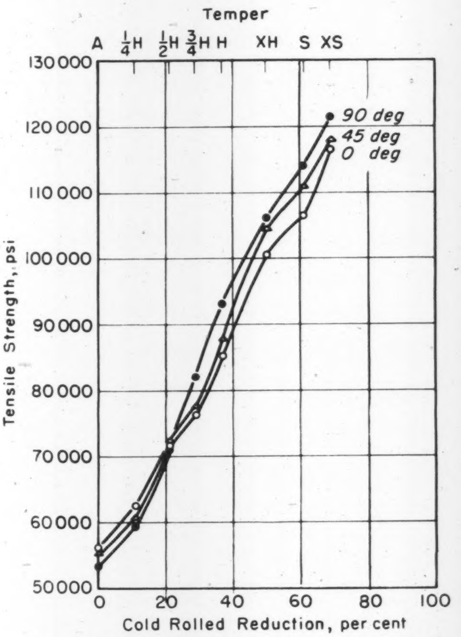

FIG. 2.—Effect of Grain Direction upon the Tensile Strength of Phosphor Bronze Grade C Strip. Lot A.

used to evaluate forming characteristics: (1) the minimum punch radius without cracking, and (2) the minimum punch radius without orange peel. A specimen of reduced formability will crack when formed over the sharp radius. This tendency decreases as the punch radius is increased until only orange peel can be observed. Further increases in punch radius will lessen the tendency to produce orange peel. Following bending, specimens were inspected macroscopically at a magnification of 7 ×.

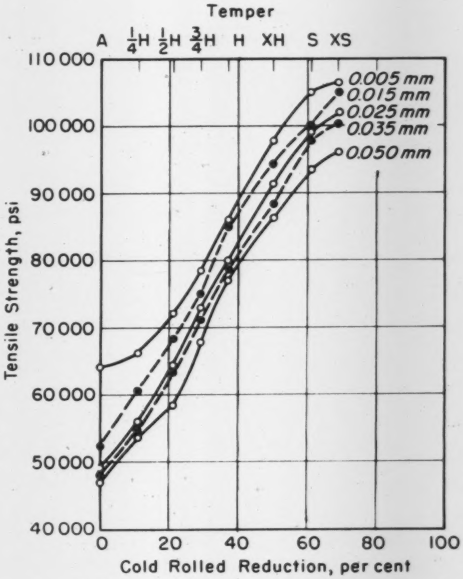

FIG. 3.—Influence of Grain Size upon Longitudinal (0 deg) Tensile Strength of Phosphor Bronze Grade A Strip. Lot B.

TEST DATA

Table III includes bend test, hardness, tensile and ductility data for various tempers, thicknesses, grain sizes and angles with rolling direction. Reported values are the averages of three to six tests.

Figure 1 illustrates the effects of grain direction and cold-rolled reduction upon

TABLE III.—TEST RESULTS.

Temper	Thickness, in.	Lot	Grain Size, mm	Minimum Punch Radius, in.[a]						Tensile Strength, psi			Elongation in 2 in., per cent			Hardness	
				Without Orange Peel			Without Cracking										
				0 deg	45 deg	90 deg	0 deg	45 deg	90 deg	0 deg	45 deg	90 deg	0 deg	45 deg	90 deg	15T	30T
Beryllium Copper—Alloy 25																	
A	0.058	A	0.035	S	S	S	S	S	S	69 800	68 600	68 500	40	48	43	77.0	51.0
	0.032	B	0.010	S	S	S	S	S	S	73 500	70 000	71 500	45	49	48	79.5	54.0
			0.020	S	S	S	S	S	S	71 600	70 600	70 500	42	53	38	78.5	52.0
			0.030	S	S	S	S	S	S	71 000	69 600	71 000	45	45	44	78.0	51.0
			0.050	S	S	S	S	S	S	69 000	66 800	68 600	43	50	48	75.0	48.5
			0.070	S	S	S	S	S	S	65 700	60 600	63 900	50	29	32	75.0	43.0
¼ H	0.051	A	0.035	S	S	S	S	S	S	82 300	80 800	82 700	25	25	19	89.0	76.0
	0.0285	B	0.010	S	S	S	S	S	S	84 700	81 000	81 500	22	32	22	89.0	75.0
			0.020	S	S	S	S	S	S	81 000	82 300	82 200	35	25	31	88.0	73.0
			0.030	S	S	S	S	S	S	80 700	79 600	80 600	35	32	28	88.0	72.5
			0.050	S	S	S	S	S	S	78 900	77 400	75 700	31	34	31	87.5	71.5
			0.070	S	S	S	S	S	S	72 600	70 300	73 300	36	31	34	87.0	69.0
½ H	0.045	A	0.035	0.010	0.015	0.020	S	S	0.005	93 400	92 500	96 800	14	11	10	92.0	80.5
	0.0254	B	0.010	0.010	0.015	0.030	S	S	0.010	99 000	96 200	98 000	10	10	8	91.0	79.5
			0.020	S	S	S	S	S	S	90 000	88 000	90 600	21	20	20	91.0	78.0
			0.030	S	S	S	S	S	S	89 700	89 000	90 500	19	16	14	90.5	78.0
			0.050	S	S	S	S	S	S	89 100	89 000	92 100	16	10	11	90.0	77.5
			0.070	S	S	S	S	S	S	81 900	87 800	85 300	27	17	21	89.5	74.0
	0.014	C	0.015	0.015	0.015	0.020	S	S	S	93 500	93 000	94 000	14	11	12	89.0	75.5
	0.008	C	0.030	0.005	0.005	0.005	S	S	S	94 100	94 400	96 700	14	10	7	90.5	79.0
H	0.036	A	0.035	0.050	0.078	0.078	0.015	0.078	0.078	113 000	110 500	117 000	5	5	5	93.5	83.5
	0.020	B	0.010	0.030	0.050	0.078	0.005	0.040	0.078	115 000	117 000	123 500	3	4	4	93.0	82.5
			0.020	0.015	0.040	0.058	S	0.015	0.050	111 500	111 000	117 500	6	7	7	92.5	82.0
			0.030	0.015	0.040	0.050	S	0.015	0.050	110 000	110 500	114 500	7	6	7	92.5	81.5
			0.050	0.015	0.040	0.050	S	0.010	0.040	108 500	104 000	112 500	3	3	4	92.0	79.5
			0.070	0.015	0.040	0.050	S	0.005	0.040	105 000	105 500	112 500	6	6	7	91.5	79.0
	0.010	C	0.010	0.010	0.020	0.020	S	0.005	0.010	111 500	111 500	118 500	3	2	4	90.0	78.5
	0.005	C	0.010	0.005	0.005	0.005	S	S	S	106 000	105 500	108 000	5	6	6	91.5	80.5
XH	0.032	A	0.035	0.063	0.093	0.109	0.050	0.078	0.109	122 000	125 000	127 500	2	4	4	94.5	85.5

BERYLLIUM COPPER—ALLOY 165

A	0.050	A	0.025	S	S	S	S	S	S	70 200	68 300	68 900	36	37	33	80.5	55.5
¼ H	0.045	A	0.025	S	S	S	S	S	S	77 200	74 900	75 200	31	35	30	86.5	71.0
½ H	0.040	A	0.025	S	0.030	0.040	S	0.005	0.020	93 200	91 300	93 200	17	11	11	91.0	80.5
H	0.032	A	0.025	0.040	0.078	0.093	0.040	0.078	0.093	113 000	114 000	117 000	4	3	5	93.5	84.0

BERYLLIUM COPPER—ALLOY 10

A	0.050	A	0.015	S	S	S	S	S	S	47 500	46 900	48 300	32	34	32	72.0	35.0
¼ H	0.045	A	0.015	S	S	S	S	S	S	54 200	53 100	54 000	20	19	17	83.5	63.0
½ H	0.040	A	0.015	0.010	0.010	0.010	S	S	S	59 000	58 300	61 400	6	7	5	86.0	68.0
	0.020	C	0.015	0.015	0.015	0.015	S	S	S	64 900	62 700	65 500	3	4	2	82.5	62.0
	0.008	C	0.015	0.005	0.005	0.005	S	S	S	67 200	66 800	70 800	1	1	1	86.5	68.5
H	0.032	A	0.015	0.015	0.015	0.020	S	S	S	71 400	67 100	68 400	4	2	2	88.0	71.5
	0.020	C	0.015	0.040	0.063	0.063	S	S	S	70 000	68 500	74 000	2	2	2	88.0	70.0
XH	0.0254	A	0.015	0.015	0.015	0.030	S	S	0.005	72 300	72 700	75 300	2	2	2	89.5	72.5
AT	0.050	A	0.015	0.093	0.093	0.093	0.093	0.093	0.093	103 800	98 500	102 300	15	15	18	90.0	80.0
½ HT	0.040	A	0.015	0.093	0.093	0.093	0.093	0.093	0.093	110 300	102 300	110 500	12	14	11	92.0	82.0
HT	0.032	A	0.015	0.078	0.078	0.093	0.078	0.078	0.093	115 500	108 000	114 000	7	5	8	93.0	83.5
	0.004	C	0.015	0.005	0.015	0.020	0.005	0.015	0.020	156 000	154 000	160 300	1	1	1	93.5	86.5

PHOSPHOR BRONZE—GRADE A

A	0.050	A	0.025	S	S	S	S	S	S	48 200	49 200	49 100	51	52	52	73.0	37.5
	0.032	B	0.005	S	S	S	S	S	S	64 300	63 200	63 400	48	47	48	70.0	35.5
			0.015	S	S	S	S	S	S	52 400	51 600	51 200	43	46	44	70.0	35.5
			0.025	S	S	S	S	S	S	49 200	49 400	49 100	48	49	47	66.0	30.0
			0.035	S	S	S	S	S	S	48 800	48 500	48 600	49	49	50	65.0	26.0
			0.050	S	S	S	S	S	S	47 100	46 400	47 200	50	53	52	64.5	25.0
¼ H	0.045	A	0.025	S	S	S	S	S	S	52 600	53 900	52 000	37	38	37	83.0	62.0
	0.0285	B	0.005	S	S	S	S	S	S	66 300	61 500	62 700	25	23	14	88.5	67.0
			0.015	S	S	S	S	S	S	60 600	58 100	59 000	26	24	22	85.0	64.0
			0.025	S	S	S	S	S	S	56 000	54 600	56 200	32	30	23	85.0	63.5
			0.035	S	S	S	S	S	S	54 900	54 400	55 000	34	29	30	84.5	62.5
			0.050	S	S	S	S	S	S	53 900	53 700	54 100	34	27	25	84.5	62.0
		C	...	S	S	S	S	S	S	53 500	53 200	53 000	33	34	33	81.5	60.0

[Continued on pp. 812 to 817.]

TABLE III.—*Continued.*

Temper	Thickness, in.	Lot	Grain Size, mm	Minimum Punch Radius, in.[a]						Tensile Strength, psi			Elongation in 2 in., per cent			Hardness	
				Without Orange Peel			Without Cracking										
				0 deg	45 deg	90 deg	0 deg	45 deg	90 deg	0 deg	45 deg	90 deg	0 deg	45 deg	90 deg	15T	30T
PHOSPOR BRONZE—GRADE A.—*Continued.*																	
½ H	0.040	A	0.025	S	S	S	S	S	S	63 300	62 700	60 600	14	22	16	87.0	70.5
	0.0285	C	0.015	S	S	S	S	S	S	63 000	63 500	64 000	20	13	13	87.5	68.5
	0.0254	B	0.005	S	S	0.005	S	S	S	72 400	71 000	73 000	13	9	7	89.0	70.5
			0.015	S	S	S	S	S	S	68 500	68 300	66 200	14	9	8	88.5	68.5
			0.025	S	S	S	S	S	S	64 500	67 300	64 700	19	10	11	88.0	68.0
			0.035	S	S	S	S	S	S	64 500	64 900	62 500	18	12	12	88.0	68.0
			0.050	S	S	S	S	S	S	58 500	60 500	60 000	29	18	13	87.0	67.5
	0.007	C	0.010	S	S	S	S	S	S	63 400	59 300	60 200	19	20	18	84.0	65.5
¾ H	0.036	A	0.025	0.015	0.030	0.030	S	S	S	70 500	68 900	68 000	10	14	12	88.5	72.0
	0.0226	B	0.005	S	S	0.015	S	S	S	79 500	78 600	82 600	7	6	4	90.5	73.5
			0.015	S	S	0.010	S	S	S	75 200	76 500	78 900	8	7	4	89.0	71.5
			0.025	S	S	0.005	S	S	S	73 000	72 400	74 100	13	8	8	89.0	71.5
			0.035	S	S	S	S	S	S	71 300	70 300	72 300	8	6	7	89.0	71.5
			0.050	S	S	S	S	S	S	67 900	69 800	71 100	13	10	10	88.0	70.0
	0.005	C	0.010	S	S	S	S	S	S	71 500	69 000	75 000	9	11	8	87.5	71.0
H	0.032	A	0.025	0.040	0.050	0.063	S	S	0.040	79 200	76 600	75 500	9	10	8	90.0	76.0
	0.020	B	0.005	0.005	0.015	0.050	S	S	0.020	86 000	85 000	82 700	5	4	3	91.0	75.5
			0.015	S	0.015	0.040	S	S	0.030	85 500	86 500	90 900	5	5	4	90.5	74.5
			0.025	S	0.015	0.030	S	S	0.010	79 800	79 000	73 700	6	..	6	90.0	74.0
			0.035	S	0.015	0.020	S	S	0.015	78 900	78 500	75 700	5	4	6	89.0	73.5
			0.050	S	0.010	0.020	S	S	0.020	77 000	79 400	84 700	7	6	7	88.5	73.0
	0.010	C	0.010	S	S	S	S	S	S	75 200	73 100	71 000	12	10	11	85.0	69.0
XH	0.0254	A	0.025	0.050	0.078	0.093	S	0.010	0.063	94 200	87 700	89 400	6	6	4	91.0	78.0
	0.0159	B	0.005	0.015	0.040	0.078	S	0.020	0.063	98 000	98 500	101 500	1	1	1	91.0	76.5
			0.015	S	0.020	0.050	S	0.010	0.050	94 500	95 900	102 000	1	2	2	90.5	76.0
			0.025	S	0.015	0.050	S	S	0.040	91 300	90 100	87 100	2	3	2	90.0	76.0
			0.035	S	0.015	0.050	S	S	0.040	88 500	93 400	95 800	3	4	4	89.5	76.0
			0.050	S	0.015	0.050	S	S	0.040	86 300	88 500	89 500	5	4	4	89.0	75.5

PHOSPHOR BRONZE GRADE A.—*Continued.*

S	0.020	A	0.025	0.063	0.093	0.109	S	0.030	0.078	102 000	97 000	97 800	4	2	2	91.0	78.0
	0.0159	C	0.015	0.015	0.020	0.040	S	S	0.030	95 500	99 900	102 500	3	3	5	90.0	75.0
	0.0126	B	0.005	0.015	0.040	0.078	S	0.015	0.063	105 000	108 500	115 000	1	2	3	90.5	79.0
			0.015	0.010	0.030	0.063	S	0.020	0.050	100 000	103 000	106 000	2	2	2	89.5	77.5
			0.025	0.005	0.020	0.063	S	0.005	0.040	99 500	102 000	104 500	2	2	4	89.5	77.0
			0.035	S	0.015	0.063	S	S	0.040	98 400	102 800	105 800	2	2	3	89.5	77.0
			0.050	S	0.010	0.063	S	S	0.040	93 500	96 700	98 300	2	2	4	89.5	76.0
	0.010	C	0.010	0.010	0.015	0.020	S	S	0.010	96 400	97 500	101 500	3	2	2	89.0	75.5
	0.005	C	0.010	0.005	0.010	0.010	S	S	0.005	93 800	101 000	101 500	1	3	4	91.0	78.5
XS	0.0159	A	0.025	0.063	0.093	0.156	S	0.040	0.078	108 500	96 500	103 000	4	2	2	91.0	78.5
	0.010	B	0.005	0.015	0.050	0.078	S	0.020	0.063	106 500	111 000	117 500	1	1	1	90.5	79.5
			0.015	0.015	0.030	0.078	S	0.005	0.050	105 000	107 000	112 500	2	1	2	90.5	79.0
			0.025	0.010	0.020	0.078	S	0.005	0.063	102 300	107 500	111 000	2	2	2	90.5	78.5
			0.035	0.005	0.020	0.078	S	0.010	0.063	100 300	105 500	109 500	2	2	3	90.0	78.0
			0.050	0.005	0.020	0.078	S	0.010	0.063	96 200	103 000	109 000	2	2	4	89.0	77.5

PHOSPHOR BRONZE—GRADE C

A	0.050	A	0.050	S	S	S	S	S	S	56 200	55 400	53 200	71	67	52	73.0	36.5
¼ H	0.045	A	0.050	S	S	0.005	S	S	S	62 500	60 800	59 900	55	53	47	84.0	64.5
½ H	0.040	A	0.050	S	0.005	0.020	S	S	S	71 900	72 400	71 000	39	33	27	89.0	74.5
	0.010	C	0.015	S	S	S	S	S	S	83 500	83 700	82 000	26	22	27	86.5	71.5
¾ H	0.036	A	0.050	0.010	0.040	0.050	S	S	0.030	76 400	77 600	82 200	29	22	20	89.5	75.5
	0.014	C	0.025	0.010	0.015	0.015	S	S	S	81 100	83 100	83 100	19	12	13	87.5	72.0
H	0.032	A	0.050	0.030	0.050	0.063	S	0.005	0.063	85 100	87 900	93 100	19	14	17	92.0	79.5
	0.008	C	0.025	S	0.005	0.005	S	S	S	92 800	95 200	94 400	14	11	12	89.0	77.0
XH	0.0254	A	0.050	0.040	0.063	0.109	S	0.050	0.109	100 500	104 500	106 000	9	8	10	92.0	80.0
	0.0159	C	...	0.010	0.030	0.063	S	0.015	0.050	109 000	111 500	116 500	3	4	6	83.0	82.0
	0.005	C	0.025	S	0.005	0.010	S	S	S	101 500	104 000	106 300	8	5	9	91.0	78.5
S	0.020	A	0.050	0.040	0.063	0.109	S	0.063	0.109	106 500	111 000	114 000	3	6	8	92.5	80.5
	0.020	C	0.035	0.030	0.078	0.156	0.015	0.063	0.125	113 500	116 500	124 000	2	3	6	93.0	81.0
	0.008	C	0.015	0.010	0.015	0.020	S	0.005	0.020	117 500	118 800	123 500	2	3	6	90.5	80.5
XS	0.0226	C	0.035	0.050	0.109	0.109	0.010	0.050	0.093	109 500	114 500	117 000	3	5	6	92.5	81.0
	0.0159	A	0.050	0.040	0.063	0.125	0.005	0.063	0.125	116 500	118 500	121 500	2	5	6	93.5	83.0
	0.010	C	0.025	0.015	0.050	0.093	S	0.030	0.093	126 000	131 000	134 000	1	2	3	92.0	82.0
	0.006	C	0.015	0.005	0.015	0.020	S	S	0.015	120 500	124 000	128 500	1	2	...	92.0	81.5

[Continued on pp. 814 to 817.]

TABLE III.—*Continued.*

Temper	Thickness, in.	Lot	Grain Size, mm	Minimum Punch Radius, in.[a]						Tensile Strength, psi			Elongation in 2 in., per cent			Hardness	
				Without Orange Peel			Without Cracking										
				0°	45°	90°	0°	45°	90°	0°	45°	90°	0°	45°	90°	15T	30T
PHOSPHOR BRONZE—GRADE D																	
A	0.050	A	0.050	S	S	S	S	S	S	65 900	64 500	57 400	59	56	32	77.5	47.0
¼ H	0.045	A	0.050	S	S	S	S	S	S	74 800	74 000	73 500	46	40	26	87.5	71.0
½ H	0.040	A	0.050	0.005	0.030	0.050	S	0.005	0.050	83 100	87 000	87 300	28	22	21	90.0	77.0
¾ H	0.036	A	0.050	0.015	0.050	0.063	S	0.020	0.063	96 200	100 500	96 200	20	12	13	92.5	81.0
0	0.005	C	. . .	S	S	0.005	S	S	S	93 300	97 200	98 700	23	12	20	91.0	78.5
H	0.032	A	0.050	0.030	0.050	0.078	S	0.040	0.078	101 500	103 500	106 800	13	11	11	93.0	82.0
XH	0.0254	A	0.050	0.030	0.063	0.109	0.005	0.063	0.109	113 000	115 500	117 500	7	7	9	93.5	83.0
S	0.020	A	0.050	0.030	0.078	0.125	0.015	0.078	0.125	120 000	117 000	124 000	3	5	7	94.5	83.5
	0.020	C	. . .	0.030	0.078	0.156	0.010	0.063	0.156	120 000	122 000	130 000	2	4	5	94.0	83.5
	0.016	C	0.035	0.020	0.040	0.063	S	0.020	0.063	114 000	117 000	119 500	3	3	8	92.5	81.0
	0.010	C	0.015	0.010	0.015	0.020	S	0.005	0.020	118 000	119 000	122 000	4	5	7	89.5	78.0
XS	0.0159	A	0.050	0.040	0.078	0.156	0.015	0.078	0.156	123 000	125 500	135 500	2	3	4	94.5	83.5
12 PER CENT NICKEL-SILVER (12—31.5—56.5)																	
A	0.050	A	0.035	S	S	S	S	S	S	60 300	61 500	62 500	41	42	47	78.0	50.5
¼ H	0.045	A	0.035	S	S	S	S	S	S	68 400	68 000	70 100	34	36	35	86.0	69.5
½ H	0.040	A	0.035	0.010	0.010	0.010	S	S	S	77 200	76 000	79 700	11	11	12	89.0	76.0
¾ H	0.036	A	0.035	0.030	0.020	0.020	S	S	S	84 300	81 300	84 800	8	7	8	90.0	77.0
	0.020	C	. . .	0.015	0.010	0.015	S	S	S	91 500	85 000	91 000	7	7	5	91.5	77.0
H	0.032	A	0.035	0.040	0.063	0.040	S	S	S	91 900	87 100	89 400	4	5	7	90.5	78.0
XH	0.0254	A	0.035	0.063	0.063	0.063	S	S	0.020	106 500	104 000	108 500	2	2	3	91.5	80.0
S	0.020	A	0.035	0.063	0.078	0.078	S	0.010	0.050	111 000	111 000	114 000	3	2	2	92.0	80.0
	0.0126	C	0.020	0.015	0.040	0.063	S	0.020	0.040	116 500	121 000	124 500	1	2	2	92.0	79.5
	0.007	C	0.015	0.020	0.040	0.063	S	0.005	0.015	123 500	121 500	126 500	1	2	2	91.0	81.0
	0.005	C	0.010	0.005	0.020	0.050	S	S	0.010	125 000	132 500	134 500	1	1	1	92.0	82.0

18 PER CENT NICKEL-SILVER (18—27—55)

A	0.050	A	0.045	S	S	S	S	S	S	62 800	60 700	60 400	42	30	37	77.5	44.5
	0.032	B	0.005	S	S	S	S	S	S	73 000	71 900	71 900	35	30	27	84.0	61.0
			0.015	S	S	S	S	S	S	67 500	69 900	66 000	25	36	13	82.5	59.5
			0.025	S	S	S	S	S	S	65 300	63 300	61 500	45	41	37	75.0	46.5
			0.035	S	S	S	S	S	S	...	...	...	..	..	..	..	..
¼ H	0.045	A	0.045	S	S	S	S	S	S	71 000	67 900	68 300	32	33	24	86.0	68.0
	0.0285	B	0.005	S	S	S	S	S	S	82 300	78 200	80 000	21	16	16	90.0	75.5
			0.015	S	S	S	S	S	S	79 400	76 200	...	23	23	..	88.5	71.5
			0.025	S	S	S	S	S	S	72 000	69 500	70 200	31	32	22	87.0	69.5
			0.035	S	S	S	S	S	S	72 000	69 000	69 500	32	32	25	87.0	68.0
½ H	0.040	A	0.045	S	S	S	S	S	S	79 100	74 000	77 000	17	18	17	89.0	75.5
	0.0254	B	0.005	0.005	S	0.005	S	S	S	93 200	88 400	92 000	8	7	6	91.0	78.5
			0.015	S	S	S	S	S	S	86 800	83 600	86 100	10	8	7	90.5	77.5
			0.025	S	S	S	S	S	S	80 800	75 500	77 300	16	13	13	89.0	74.0
			0.035	S	S	S	S	S	S	78 200	74 700	76 000	21	21	20	88.0	72.5
	0.020	C	0.010	S	S	0.005	S	S	S	87 000	81 000	85 000	11	8	7	90.0	75.0
¾ H	0.036	A	0.045	0.020	0.005	0.010	S	S	S	88 000	82 300	84 500	8	8	7	90.5	78.5
	0.0226	B	0.005	0.015	0.005	0.015	S	S	0.005	99 900	95 800	99 800	2	3	3	91.5	79.0
			0.015	0.015	0.005	0.005	S	S	S	95 500	90 900	94 800	4	3	3	91.0	78.5
			0.025	0.015	S	0.005	S	S	S	91 300	88 400	90 400	5	5	6	91.0	77.0
			0.035	0.015	S	S	S	S	S	88 400	83 700	86 500	7	8	7	91.0	76.0
H	0.032	A	0.045	0.030	0.020	0.030	S	S	0.015	94 100	88 000	90 600	3	4	3	91.0	79.5
	0.020	B	0.005	0.015	0.020	0.030	S	0.005	0.030	104 500	106 800	110 500	2	3	2	92.0	79.0
			0.015	0.015	0.015	0.020	S	S	S	100 800	98 100	101 800	2	3	2	92.0	79.0
			0.025	0.015	0.005	0.015	S	S	S	...	...	...	...	...	...	...	...
			0.035	0.015	S	0.010	S	S	0.005	95 800	89 800	92 800	4	6	4	92.0	79.0
XH	0.0254	A	0.045	0.030	0.030	0.040	S	S	0.030	100 500	97 000	...	1	2	...	91.5	80.0
	0.0159	B	0.005	0.015	0.020	0.050	S	0.010	0.050	110 900	110 000	118 800	1	1	1	92.5	80.5
			0.015	0.015	0.020	0.040	S	S	0.030	110 000	114 000	117 000	1	2	2	92.0	80.5
			0.025	0.015	0.015	0.020	S	S	0.015	102 000	98 900	106 400	1	2	1	92.0	79.0
			0.035	0.015	0.015	0.020	S	S	0.015	92 200	92 800	100 800	2	4	2	91.0	78.0
S	0.020	A	0.045	0.030	0.040	0.050	S	S	0.040	107 000	104 500	110 500	2	3	2	91.5	79.5
	0.015	C	0.025	0.015	0.040	0.078	S	0.020	0.063	121 500	111 500	115 500	2	1	1	92.0	80.0
	0.0126	B	0.005	0.020	0.030	0.063	S	0.015	0.063	118 500	115 000	124 200	1	1	1	92.0	81.0
			0.015	0.015	0.020	0.050	S	0.015	0.040	115 800	118 000	118 800	1	1	1	92.0	81.0
			0.025	0.015	0.020	0.030	S	S	0.020	106 800	109 300	113 200	1	1	1	91.5	79.0
			0.035	0.015	0.020	0.030	S	S	0.020	99 600	102 000	104 000	3	4	4	91.0	79.0

[Continued on pp. 816 to 817.]

TABLE III.—*Continued.*

Temper	Thickness, in.	Lot	Grain Size, mm	Minimum Punch Radius, in.[a]						Tensile Strength, psi			Elongation in 2 in., per cent			Hardness	
				Without Orange Peel			Without Cracking										
				0°	45°	90°	0°	45°	90°	0°	45°	90°	0°	45°	90°	15T	30T
	0.007	C	0.010	0.020	0.020	0.040	S	0.015	0.020	124 500	129 500	131 500	1	2	2	91.0	81.0
XS	0.010	B	0.005	0.020	0.040	0.063	S	0.015	0.063	126 000	129 000	132 500	1	1	1	91.5	81.5
			0.015	0.015	0.030	0.063	S	0.020	0.050	121 800	127 500	132 300	1	1	1	91.5	81.5
			0.025	0.015	0.030	0.050	S	S	0.020	119 700	116 800	123 500	1	1	1	91.0	81.0
			0.035	0.015	0.020	0.040	S	S	0.020	116 500	...	119 600	1	..	2	90.5	80.0
GILDING (95-5)																	
A	0.050	A	0.035	S	S	S	S	S	S	33 200	33 100	33 600	42	44	42	55.0	11.0
¼ H	0.045	A	0.035	S	S	S	S	S	S	37 500	38 400	38 500	37	35	35	77.0	46.5
½ H	0.040	A	0.035	S	S	S	S	S	S	45 600	44 600	45 600	8	9	11	82.0	61.5
¾ H	0.036	A	0.035	0.005	0.005	0.005	S	S	S	49 400	49 000	49 900	4	3	7	83.0	61.5
H	0.032	A	0.035	0.010	0.010	0.010	S	S	S	51 500	50 600	51 400	3	3	3	84.5	62.0
XH	0.0254	A	0.035	0.020	0.020	0.020	S	S	S	58 700	55 500	56 200	2	2	2	85.0	63.0
S	0.020	A	0.035	0.020	0.020	0.020	S	S	S	63 100	60 300	61 300	2	2	2	85.0	64.0
XS	0.0159	A	0.035	0.030	0.040	0.050	S	S	S	67 400	64 100	64 400	2	2	2	85.0	64.5
BRASS (70-30)																	
A	0.050	A	0.070	S	S	S	S	S	S	43 300	43 900	45 300	47	52	49	76.5	48.5
	0.032	B	0.005	S	S	S	S	S	S	60 900	61 200	60 200	33	29	27	81.5	58.5
			0.015	S	S	S	S	S	S	56 100	59 600	59 200	47	37	34	78.0	52.0
			0.025	S	S	S	S	S	S	55 300	55 000	53 900	48	44	36	72.0	44.0
			0.035	S	S	S	S	S	S	49 900	45 700	49 300	53	56	50	70.0	27.0
			0.050	S	S	S	S	S	S	46 700	43 000	46 100	56	44	42	59.5	24.0
¼ H	0.045	A	0.070	S	S	S	S	S	S	51 200	49 300	52 400	40	41	43	81.5	57.0
	0.0285	B	0.005	S	S	S	S	S	S	71 700	69 300	71 800	15	11	11	88.5	70.0
			0.015	S	S	S	S	S	S	68 100	66 400	69 000	30	20	14	87.0	71.5
			0.025	S	S	S	S	S	S	60 600	55 900	60 300	34	27	21	85.5	63.5
			0.035	S	S	S	S	S	S	57 700	54 600	56 100	38	38	31	83.5	59.5
			0.050	S	S	S	S	S	S	52 400	50 300	52 600	44	45	40	80.0	53.0

½ H	0.040	A	0.070	S	S	S	S	S	S	57 300	53 700	59 000	27	26	28	85.5	67.0
	0.0254	B	0.005	S	0.015	0.015	S	S	S	84 300	91 400	92 400	6	3	3	91.0	78.5
			0.015	S	0.005	0.005	S	S	S	81 000	80 000	81 400	11	7	6	89.5	75.0
			0.025	S	0.005	0.005	S	S	S	71 300	71 400	74 000	16	9	9	89.0	71.0
			0.035	S	S	S	S	S	S	63 900	64 500	67 200	22	12	11	87.5	67.5
			0.050	S	S	S	S	S	S	63 500	59 500	62 200	24	20	17	87.0	66.5
	0.010	C	0.035	S	S	S	S	S	S	63 500	62 000	62 500	21	16	17	84.5	65.5
	0.005	C	0.040	S	S	S	S	S	S	56 900	53 000	57 100	16	14	16	83.5	61.5
¾ H	0.036	A	0.070	0.005	0.005	0.010	S	S	S	65 000	62 800	64 400	14	12	14	87.5	70.5
	0.0226	B	0.005	0.020	0.020	0.050	S	S	0.040	86 400	91 800	93 500	3	2	4	90.0	75.0
			0.015	0.005	0.015	0.040	S	S	0.020	85 100	87 700	87 300	4	4	4	90.0	74.5
			0.025	S	0.015	0.020	S	S	S	78 300	81 000	82 200	8	5	7	89.0	73.5
			0.035	S	0.010	0.020	S	S	S	72 300	71 100	75 900	12	7	9	87.5	69.5
			0.050	S	0.005	0.010	S	S	S	65 400	66 900	67 300	19	17	14	87.5	68.5
	0.020	C	0.035	0.030	0.030	0.030	S	S	S	69 500	69 000	70 000	15	11	12	87.0	69.5
	0.008	C	0.030	0.015	0.015	0.015	S	S	S	70 000	69 000	70 400	14	11	13	83.0	68.0
H	0.032	A	0.070	0.015	0.030	0.040	S	S	S	74 200	71 400	73 200	10	8	8	88.5	73.5
	0.020	B	0.005	0.020	0.050	0.063	S	0.005	0.040	97 400	94 500	100 000	3	2	2	92.0	76.5
			0.015	0.020	0.040	0.050	S	S	0.020	91 800	93 000	99 800	3	3	3	90.5	76.5
			0.025	0.020	0.020	0.040	S	S	0.020	84 300	89 700	94 000	4	3	4	90.0	75.0
			0.035	0.020	0.020	0.030	S	S	S	79 500	79 600	82 900	7	6	7	88.5	71.0
			0.050	0.010	0.015	0.020	S	S	S	74 800	73 700	78 300	8	8	8	88.0	69.0
XH	0.0254	A	0.070	0.015	0.040	0.063	S	S	0.040	85 500	80 100	80 000	8	4	4	89.5	75.0
	0.0159	B	0.005	0.015	0.050	0.078	S	0.020	0.063	103 300	104 000	101 500	2	2	2	90.0	76.5
			0.015	0.015	0.030	0.063	S	0.015	0.050	100 500	103 300	109 000	3	3	3	89.5	76.5
			0.025	0.015	0.020	0.040	S	S	0.030	93 100	92 500	91 100	2	2	4	89.5	75.0
			0.050	0.015	0.020	0.040	S	S	0.015	86 700	87 900	91 200	2	3	6	88.0	74.0
S	0.020	A	0.070	0.015	0.040	0.078	S	0.015	0.050	92 200	91 900	89 100	4	3	2	90.0	75.5
	0.0126	B	0.005	0.015	0.040	0.078	S	0.020	0.063	103 500	107 000	113 000	2	2	1	90.0	76.5
			0.015	0.015	0.030	0.063	S	0.015	0.050	103 000	102 300	107 800	2	3	2	89.0	76.5
			0.025	0.015	0.020	0.040	S	0.015	0.030	101 000	103 000	107 000	1	3	2	88.5	76.5
			0.035	0.015	0.020	0.040	S	0.005	0.020	97 000	98 500	101 000	2	2	2	88.0	75.0
			0.050	0.015	0.020	0.040	S	0.005	0.020	93 100	94 100	98 700	2	3	3	88.0	74.5
XS	0.0159	A	0.070	0.020	0.050	0.093	0.005	0.020	0.063	100 500	96 700	95 000	2	2	2	90.0	76.0
	0.010	B	0.005	0.015	0.050	0.078	S	0.020	0.063	109 300	113 500	121 800	1	2	2	89.5	78.0
			0.015	0.015	0.040	0.078	S	0.015	0.063	107 000	110 500	113 000	2	2	2	89.0	77.5
			0.025	0.015	0.020	0.063	S	0.015	0.030	105 000	108 500	110 000	2	2	2	89.0	76.5
			0.035	0.015	0.020	0.040	S	0.005	0.020	102 300	103 500	107 300	1	2	2	89.0	76.5
			0.050	0.015	0.020	0.040	S	0.005	0.020	97 300	98 200	114 000	1	2	3	89.0	76.5

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[a] S = sharp.

TABLE IV.—FORMABILITY OF SEVERAL COPPER ALLOYS IN STRIP FOR M.
Forming 90-deg bends in strip 0.010 to 0.050 in. thick[a]

	Temper	Ratio, Punch Radius to Stock Thickness[b]					
		Without Orange Peel			Without Cracking		
		0 deg	45 deg	90 deg	0 deg	45 deg	90 deg
Beryllium copper alloy 25	A	S	S	S	S	S	S
	¼ H	S	S	S	S	S	S
	½ H	0.5	0.7	1.1	S	S	S
	H	1.0	2.0	2.2	S	0.8	2.0
	XH	2.0	2.9	3.4	1.6	2.4	3.4
Beryllium copper alloy 165	A	S	S	S	S	S	S
	¼ H	S	S	S	S	S	S
	½ H	S	0.7	1.0	S	0.1	0.5
	H	1.3	2.4	2.9	1.3	2.4	2.9
Beryllium copper alloy 10	A	S	S	S	S	S	S
	¼ H	S	S	S	S	S	S
	½ H	0.4	0.4	0.4	S	S	S
	H	0.5	0.5	0.6	S	S	S
	XH	0.6	0.6	1.2	S	S	0.2
	AT	1.9	1.9	1.9	1.9	1.9	1.9
	½ HT	2.4	2.4	2.4	2.4	2.4	2.4
	HT	2.4	2.4	2.9	2.4	2.4	2.9
Phosphor bronze grade A	A	S	S	S	S	S	S
	¼ H	S	S	S	S	S	S
	½ H	S	S	S	S	S	S
	¾ H	0.2	0.4	0.5	S	S	S
	H	0.3	0.8	1.7	S	S	1.0
	XH	1.0	2.0	3.4	S	0.2	2.5
	S	1.3	2.2	5.0	S	0.4	3.0
	XS	2.5	4.0	9.0	S	1.5	5.6
Phosphor bronze grade C	A	S	S	S	S	S	S
	¼ H	S	S	0.1	S	S	S
	½ H	S	0.1	0.5	S	S	S
	¾ H	0.3	1.0	1.2	S	S	0.8
	H	0.5	1.4	2.0	S	0.2	2.0
	XH	1.2	2.2	4.0	S	2.0	4.0
	S	1.6	3.2	5.5	S	3.2	5.5
	XS	2.2	4.6	7.9	0.3	4.0	7.9
Phosphor bronze grade D	A	S	S	S	S	S	S
	¼ H	S	S	S	S	S	S
	½ H	0.1	0.8	1.3	S	0.1	1.3
	¾ H	0.4	1.4	1.8	S	0.6	1.8
	H	1.0	1.6	2.4	S	1.3	2.4
	XH	1.2	2.5	4.3	0.2	2.5	4.3
	S	1.5	4.0	7.0	0.7	3.6	7.0
	XS	2.5	4.9	9.8	1.0	4.9	9.8
Nickel-silver 12 per cent	A	S	S	S	S	S	S
	¼ H	S	S	S	S	S	S
	½ H	0.3	0.3	0.3	S	S	S
	¾ H	0.8	0.6	0.6	S	S	S
	H	1.3	2.0	1.3	S	S	S
	XH	2.5	2.5	2.5	S	S	0.8
	S	3.0	4.0	5.0	S	1.0	2.5

TABLE IV.—*Concluded.*

	Temper	Ratio, Punch Radius to Stock Thickness[b]					
		Without Orange Peel			Without Cracking		
		0 deg	45 deg	90 deg	0 deg	45 deg	90 deg
Nickel-silver 18 per cent	A	S	S	S	S	S	S
	¼ H	S	S	S	S	S	S
	½ H	S	S	S	S	S	S
	¾ H	0.6	0.2	0.3	S	S	S
	H	0.9	0.6	0.8	S	S	0.2
	XH	1.1	1.2	1.4	S	S	0.6
	S	1.3	2.0	4.0	S	0.8	2.6
	XS	1.5	3.0	5.5	S	1.0	3.0
Gilding 95-5	A	S	S	S	S	S	S
	¼ H	S	S	S	S	S	S
	½ H	S	S	S	S	S	S
	¾ H	0.1	0.1	0.1	S	S	S
	H	0.3	0.3	0.3	S	S	S
	XH	0.8	0.8	0.8	S	S	S
	S	1.0	1.0	1.0	S	S	S
	XS	1.8	2.5	3.1	S	S	S
Brass 70-30	A	S	S	S	S	S	S
	¼ H	S	S	S	S	S	S
	½ H	S	S	S	S	S	S
	¾ H	0.1	0.4	0.6	S	S	S
	H	0.7	1.3	1.6	S	S	0.5
	XH	0.8	1.4	2.5	S	S	1.8
	S	1.0	1.8	3.7	S	1.0	2.9
	XS	1.4	3.1	6.1	S	1.4	3.5

[a] Strip under 0.010 in.—use 50 to 75 per cent of above ratios.

[b] S = sharp.

the formability of phosphor bronze grade D strip. Relative formability is expressed as the ratio of punch radius to stock thickness. Therefore, a value of 2 means that the minimum safe punch radius for forming a 90-deg bend without producing orange peel would be twice the stock thickness.

The influences of grain direction, grain size, and cold reduction upon tensile strength are shown in Figs. 2 and 3.

DISCUSSION OF DATA

Formability:

As indicated in Table III, the softer tempers in thicknesses up to 0.050 in. can be cold formed to a 90-deg bend around a sharp radius without cracking or exhibiting orange peel. In the intermediate tempers, moderate radii were required to produce satisfactory bends; however, there was little indication of directionality. With a further increase in cold-rolled reductions, a pronounced directionality was evident in most of the materials. With the exception of nickel-silver, which exhibited non-uniform directionality, the forming characteristics in the transverse or 90-deg direction were substantially reduced compared with those observed in the longitudinal direction.

Some evidence of directionality can also be observed in the tensile results, although once again the nickel-silver alloys show a different pattern. Other investigators (5), in studying the tensile properties of nickel-silver strip, have found directional characteristics differing from those obtained in other copper-base products. Gilding metal, in addition to being the softest material tested, exhibited the least degree of work hardening and directionality.

TABLE V.—RELATIVE FORMABILITY OF COPPER ALLOY STRIP COMPARED WITH EARLIER INVESTIGATIONS.

Forming 90-deg bends in Strip 0.010 to 0.050 in. thick.[a]

	Temper	Ratio, Punch Radius to Stock Thickness[b]						References
		Present Investigation			Earlier Investigation			
		0 deg	45 deg	90 deg	0 deg	45 deg	90 deg	
Beryllium copper alloy 25	A	S	S	S	S	S	S	3
	¼ H	S	S	S	S	S	S	3
	½ H	0.5	0.7	1.1	3.0	3.5	4.0	3
	H	1.0	2.0	2.2	4.5	6.0	7.5	3
Phosphor bronze grade A	½ H	S	S	S	S	S	S	1
	H	0.3	0.8	1.7	0.7	1.5	3.0	1
	XH	1.0	2.0	3.4	1.5	3.0	...	1
	S	1.3	2.2	5.0	2.0	6.0	...	1
Phosphor bronze grade C	½ H	S	0.1	0.5	S	S	S	1, 2
	H	0.5	1.4	2.0	1.0	2.0	3.0	1, 2
	XH	1.2	2.2	4.0	1.2	3.0	6.0	2
	S	1.6	3.2	5.5	1.4	6.0	10.0	1, 2
	XS	2.2	4.6	7.9	1.5	6.0	11.0	1, 2
Nickel-silver 18 per cent	½ H	S	S	S	S	S	S	1, 2
	H	0.9	0.6	0.8	1.5	1.5	3.0	1, 2
	XH	1.1	1.2	1.4	2.5	3.0	5.0	1, 2
Brass 70-30	½ H	S	S	S	S	S	S	1, 2
	¾ H	0.1	0.4	0.6	S	S	S	1, 2
	H	0.7	1.3	1.6	S	S	0.7	1, 2
	XH	0.8	1.4	2.5	1.0	2.0	5.0	1, 2
	S	1.0	1.8	3.7	1.5	4.5	10.0	1, 2
	XS	1.4	3.1	6.1	1.8	6.0	...	2

[a] Strip under 0.010 in.—use 50 to 75 per cent of above ratios.

[b] S = sharp.

Table IV summarizes the bending characteristics for material ranging from 0.010 to 0.050 in. thick. In the case of thinner gages, slightly improved forming characteristics were observed, so that for strip under 0.010 in. thick values approximately 50 to 75 per cent of those given in the table can be safely employed. In calculating the formability ratios, extreme grain sizes have been excluded so that various sizes reported should prove typical for commercial strip.

Table V affords a comparison between the present investigation and similar studies of the bending characteristics of the same materials. Unfortunately, these earlier studies were not as complete with respect to temper, so that all of the tempers tested are not included in this comparison. In the case of beryllium copper (alloy 25), the improved formability reported herein agrees with the higher elongations also observed. These changes are perhaps due to the gradual decrease in beryllium content over the past few years, which would reduce the matrix strength of the alloy.

Tensile Studies:

The directionality previously noted in the bend tests was also apparent in the tensile data. Directionality was checked by testing specimens at 0, 45, and 90 deg with respect to the rolling direction. No appreciable tensile directionality was observed in the low and moderate tempers, although directional effects were readily apparent in the higher tempers. This effect is illustrated in Fig. 2 and was rather pronounced for phosphor bronze, beryllium copper, and brass. In the case of nickel-silver and gilding, the directional characteristics are not as clearly discernible.

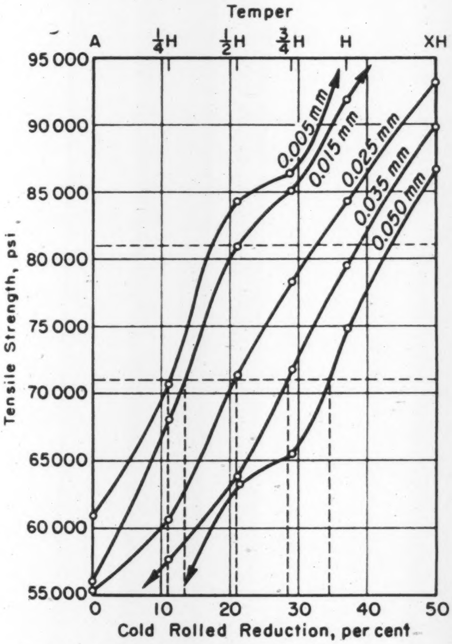

FIG. 4.—Effect of Grain Size upon the Degree of Cold Rolling Required to Produce the Hard Temper (4 B&S Numbers Hard) in 70-30 Brass. Lot B.

The influence of grain size is demonstrated in Fig. 4 for brass strip. The limiting tensile strengths for the hard temper (4 B&S Numbers Hard) are 71,000 to 81,000 psi. In the case of 0.050-mm grain size, a reduction in thickness of approximately 35 per cent is required; however, for strip with a 0.005-mm grain size, a cold reduction of only 11 per cent is needed. For intermediate grain sizes, cold reductions between these two limits can be applied to meet the minimum tensile requirements for this temper, as shown in Fig. 4.

The relative formability for the materials tested for various levels of tensile strength is illustrated in Fig. 5. Therefore, to meet a given formability requirement, beryllium copper and nickel-silver provide a substantially higher level of strength than phosphor bronze or brass. The values for beryllium copper apply for the heat-treatable condition, so that following precipitation hardening strengths approximately double those indicated would be realized. The relative formability ratios are based upon the bending characteristics in any direction.

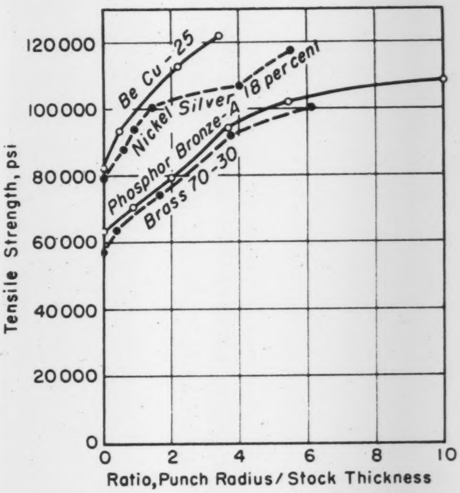

FIG. 5.—Relationship between Tensile Strength and Relative Formability of Several Copper Alloys in Strip Form for Longitudinal (0 deg) Specimens. Lot A.

SUMMARY AND CONCLUSIONS

These tests have been conducted to correlate the forming characteristics of several copper alloys in strip form with tensile and hardness data. Minimum safe forming radii to produce a 90-deg bend were obtained by a direct method employing horizontal bending around a radius block. The effects of temper, stock thickness, grain size, and grain direction were evaluated. In addition, the relative forming characteristics of these materials have been compared, so that where substitution of one alloy for another may be desired, forming characteristics for the new material can be readily estimated.

The following conclusions can be drawn:

1. Softer tempers can be bent sharp to a 90-deg angle without cracking or displaying orange peel.

2. Harder tempers require increased radii for safe bending.

3. Most of the materials tested displayed directionality as evidenced by bend and tension tests, particularly in the harder tempers.

4. Strip having relatively fine grain structure offers the best combination of formability and tensile properties.

5. Of the materials tested, beryllium copper offers the highest strength and hardness for a given level of formability—even prior to precipitation hardening.

This investigation is part of a continuing program for the evaluation of the forming characteristics of spring materials. Therefore, it is hoped that data on other alloys can be reported at a later date, and that some of the present anomalous conditions can be clarified.

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(2) G. R. Gohn, "The Forming Properties of Some Non-Ferrous Sheet Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, pp. 207–221 (1936).

(3) J. T. Richards and E. M. Smith, "The Forming Characteristics of Beryllium Copper Strip," *Proceedings*, Am. Soc. Testing Mats., Vol. 50, pp. 1085–1100 (1950).

(4) G. R. Gohn and S. M. Arnold, "Evaluation of the Forming Properties in Bending of Sheet Aluminum Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 46, pp. 1053–1061 (1946).

(5) G. R. Gohn, J. P. Guerard, and G. J. Herbert, "The Mechanical Properties of Some Nickel Silver Alloy Strips," *Proceedings*, Am. Soc. Testing Mats., Vol. 54, pp. 229–256 (1954).

EFFECT OF IRRADIATION ON THE MECHANICAL PROPERTIES OF ZIRCONIUM*

By R. S. Kemper, Jr.,[1] and W. S. Kelly[1]

Synopsis

Data are presented to show the effects of cold working and subsequent neutron irradiation on the tensile properties and hardness of arc-melted Bureau of Mines zirconium. Material in six initial states, annealed and with 10, 20, 30, 40, and 50 per cent reduction of area by cold swaging, was irradiated at 50 to 60 C (120 to 140 F) to integrated thermal flux levels of approximately 5.7×10^{19}, 1.5×10^{20}, and 2.4×10^{20} nvt. The results of post-irradiation vacuum annealing at 250, 300, and 350 C (480, 575, and 660 F) of tension specimens are given.

Increases in hardness and yield strength resulting from the irradiation were more pronounced in the annealed material and were progressively less at increasing levels of cold work. More uniform increases in ultimate strength and decreases in ductility were found. The radiation damage observed in the mechanical properties was essentially saturated at the exposure levels investigated. Slight annealing of radiation-induced increases in yield strength occurred in 100 hr at 250 C (480 F), and approximately 70 per cent of the radiation damage was removed in 160 hr at 350 C (660 F). Recovery of the radiation damage in annealed, irradiated material was greater than recovery of strain hardening in 50 per cent cold-worked, unirradiated material for the same annealing conditions.

The over-all combination of the physical, mechanical, chemical, and nuclear properties of zirconium metal and certain zirconium alloys has made their use as reactor-core structural materials attractive. One of the prime considerations in the evaluation of the material and the design of structural components must be the effects of the operating environment upon these properties. It would be desirable from the standpoint of neutron economy and material cost to utilize the work-hardening characteristics of zirconium. A number of investigations have been conducted to determine the change in density,[2, 3] elec-

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[1] Engineers, Metallurgy Research Sub-Section, Hanford Atomic Products Operation, General Electric Co., Richland, Wash.

[2] R. G. Wheeler, HW-31132, Mar. 12, 1954 (SECRET).

[3] D. O. Leeser, ANL-4792, Nov. 1953, p. 51. (SECRET).

trical resistivity,[3-7] impact strength,[8, 9] hardness,[2, 3, 5, 6, 7, 8] tensile properties,[3, 6, 10] fatigue strength,[3] and creep rate[11] of zirconium or zirconium alloys due to neutron bombardment. The major portion of these studies was made using crystal bar zirconium or zirconium alloys, and no data other than electrical resistivity, hardness, and density have been reported for material cold-worked prior to irradiation. Limited data are available from which the influence of irradiation temperature may be determined. Hardness changes resulting from post-irradiation annealing have been determined and an excellent study of the recovery of radiation damage in annealed and cold-worked crystal bar zirconium has been reported by Bleiberg and Castleman[7] using electrical resistivity measurements.

TABLE I.—ANALYSES OF INGOT.

SPECTROGRAPHIC ANALYSIS, PPM							
Al	30	Pb	50	Fe	300	V	20
Sb	10	Mg	20	Mn	10	Mo	10
Bi	1	Zn	50	Ni	5	Cd	1
Sn	13	Cr	5	Si	20	B	0.5
Cu	50	Co	10	Ti	50	Hf	81
CHEMICAL ANALYSIS, PPM							
N_2	40	C	80	O_2	1070	Fe	400

This investigation was made to determine: (*1*) the effects of neutron irradiation on the tensile properties and hardness of zirconium produced by the Kroll process, and (*2*) the influence of various degrees of prior cold work on the extent of radiation damage. Post-irradiation annealing for extended periods of time was done to estimate the effect of temperature during exposure and to determine whether there are gross differences between recovery rates for cold work and radiation damage or a combination of cold work and radiation damage.

MATERIAL

The material used was prepared from one arc-melted ingot of Bureau of Mines zirconium. The chemical and spectrographic analyses of this ingot are given in Table I.

The material was fabricated into $\frac{1}{2}$-in. diameter rods at the Bureau of Mines, Albany, Ore., according to the following schedule. The ingot was hot forged and rolled to a rod approximately 1 in. in diameter at 800 C (1472 F). This rod was machined to remove surface imperfections and cold-swaged to 0.710-in. diameter. It was then cut into six 18-in. lengths. Five of these lengths were hot swaged at 700 C (1292 F) to 0.640, 0.600, 0.560, 0.525, and 0.500 in. in diameter, respectively. All six rods were then annealed 15 min. at 700 C (1292 F). Five of the rods were cold swaged to $\frac{1}{2}$-in. diameter resulting in material with 9.1, 20.1, 30.6, 39.1, and 50.4 per cent cold reduction.

SPECIMEN AND IRRADIATION DETAILS

The tension specimens employed were standard ASTM $\frac{1}{4}$-in. diameter,[12] 1-in. gage length specimens, and the wafers used for hardness and metallographic studies were $\frac{7}{16}$-in. diameter, $\frac{1}{4}$-in. thick cylinders. One wafer and one specimen were placed in each of three holes of a 2S aluminum container under an atmosphere of helium and the cap was tightly seated. The caps were later heli-arc

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[4] M. L. Bleiberg, R. L. Ely. Jr., and W. F. Witzig, "Correlation of Zirconium Electrical Resistivity with Fast Flux," WAPD-77, May 20, 1953 (DECLASSIFIED).

[5] M. L. Bleiberg and L. S. Castleman, WAPD-78, Mar. 17, 1953 (CONFIDENTIAL).

[6] J. D. Eichenberg and L. S. Castleman, WAPD-90, Nov. 5, 1953 (SECRET).

[7] M. L. Bleiberg and L. S. Castleman, WAPD-107, Apr. 23, 1954 (CONFIDENTIAL).

[8] M. L. Bleiberg, WAPD-MDM-10, May 19, 1954 (SECRET).

[9] J. J. Cadwell, HW-37979, Jul. 1955 (SECRET).

[10] D. O. Leeser, "Evaluating Basic Materials for Nuclear-Fueled Power Plants," *Mechanical Engineering*, Vol. 77, No. 6, June 1955, p. 501.

[11] R. H. Fillnow, L. A. Cook, and W. E. Johnson, WAPD-63, May 19, 1953 (SECRET).

[12] Tentative Methods of Tension Testing of Metallic Materials (E 8 – 54 T), 1955 Book of ASTM Standards, Part I, p. 1551.

welded with the lower part of the container immersed in a dry-ice water bath to avoid heating the specimens.

Two tension specimens and two wafers from each cold-work level were charged at each of three exposures for a total irradiation of 36 tension specimens and 36 wafers.

The irradiations were carried out in three separate water-cooled, central zone, process channels of a Hanford reactor. The charging pattern was identical for the three exposures with the four specimen containers centered front to rear with normal fuel pieces and spaced with one fuel piece between each container. The exposures reported were obtained from the average heat generation of the surrounding fuel channels and an assumed cosine flux distribution along the tube.[13]

TABLE II.—POLISH-ETCH SOLUTIONS FOR ZIRCONIUM.[a]

Solution A	45 ml $C_3H_5(OH)_3$
	45 ml HNO_3 (Conc.)
	8–10 ml HF (48 per cent)
Solution B	45 ml H_2O
	45 ml HNO_3 (Conc.)
	8–10 ml HF (48 per cent)

[a] J. M. Cain, Jr., "A Simplified Procedure for Metallography of Zirconium and Hafnium and Their Alloys," *Zirconium and Zirconium Alloys*, Am. Soc. Metals, pp. 176–185.

Heat generation and heat transfer calculations that were made for the assembly predicted a maximum specimen temperature of 50 to 60 C (120 to 140 F) during exposure.

Test Procedure

Rockwell hardness readings were taken on one side of each wafer before canning. The opposite side of each wafer was prepared for metallographic examination by polishing through No. 000 emery paper. One of the chemical polish-etch solutions given in Table II was then used and representative areas were photographed at 250 ×. This structure could be seen in most cases after exposure, but scratches and foreign material on the surface picked up during remote handling made it necessary to repolish the specimens. This was accomplished by lapping on a rotary wheel with 600 mesh grit cloth. The surface was swabbed with the polish-etch solution after lapping and the structure examined.

The hardness of the irradiated samples was determined on a remotely-operated Rockwell hardness testing machine. Several readings were taken on each wafer adjacent to the pre-irradiation indents.

The tensile properties were determined for the control material using a Baldwin 60,000-lb testing machine. Stress-strain curves were obtained employing a 1-in. gage length microformer-type extensometer and a Baldwin stress-strain recorder. Tests on the irradiated material were conducted on a lead-shielded Tinius-Olsen 60,000-lb testing machine. A microformer-type extensometer was remotely attached to the specimen, and the stress-strain curve was obtained in the normal manner on a Tinius-Olsen recorder. Strain was recorded to approximately 0.100 in per in., which in some cases provided a complete curve to fracture and for all the specimens provided data to the end of uniform strain.

The 1-in. gage length was marked on the specimens before testing with a gage point punch. After fracture, the lengths from the gage marks on each side of the specimen to points at the fracture for each specimen half were measured optically with a comparator. The total elongations reported were determined from these data. The reduction of area after fracture was determined by measuring several diameters at the fracture surface for each specimen-half. These measurements were also made using the comparator which was calibrated by measuring a micrometer standard.

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[13] W. H. Clark, HW-33891, Nov. 24, 1954 (SECRET).

Annealing of the tensile samples was accomplished in an evacuated tube furnace[14] situated inside a cell in the Radiometallurgy Laboratory. A dynamic vacuum of approximately 10^{-4} mm of mercury was maintained during the annealing period. Six specimens were placed within the center 4-in. of the furnace length and heated in one furnace loading. This central portion of the furnace was held within ± 5 C of the desired temperature as indicated by a thermocouple located inside the evacuated tube.

METALLOGRAPHIC EXAMINATION

The microstructure of surfaces transverse to the rolling direction for the 36 wafers included in the test was examined before and after exposure. The examinations at 250 × did not reveal any changes due to the neutron irradiation or to thermal conditions existing during the exposure. The average grain diameter of the annealed material was approximately 0.02 mm. Examination of sections parallel to the rolling direction for the higher levels of cold work revealed the normal elongated grain structure of highly cold-worked material.

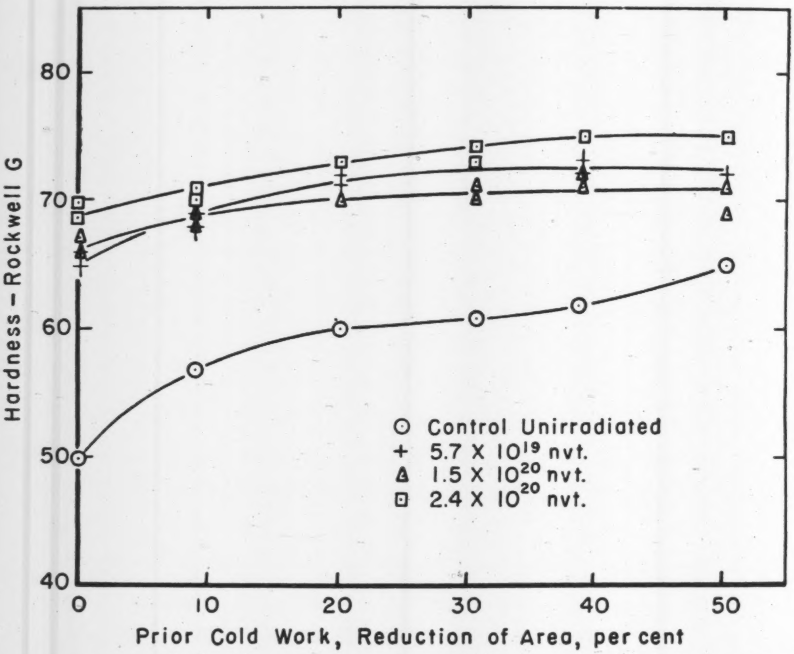

FIG. 1.—Hardness Changes Resulting from Irradiation of Arc-Melted Bureau of Mines Zirconium.

Exposure Temperature—50 to 60 C

HARDNESS

The hardness data obtained are shown in Fig. 1. At least four readings were taken on each wafer. The plotted values are the average of these readings with the variation indicated by the size of the plotted points. The initial change in hardness was greatest in the annealed material and was progressively less for increasing levels of prior cold work. This hardness change in the annealed material was as large as that produced by cold working the material to 50 per cent reduction of area. The hardness after the second exposure was essentially the same as after the initial exposure, but definitely increased again at all levels of cold work, after the longest irradiation. The data for samples irradiated to 2.4 × 10^{20} nvt indicate that the radiation-hardening approaches a saturation level for this exposure temperature at which the hardness is essentially independent of the prior cold work.

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[14] W. S. Kelly and R. E. Hueschen, HW-33944, Dec. 1, 1954 (SECRET).

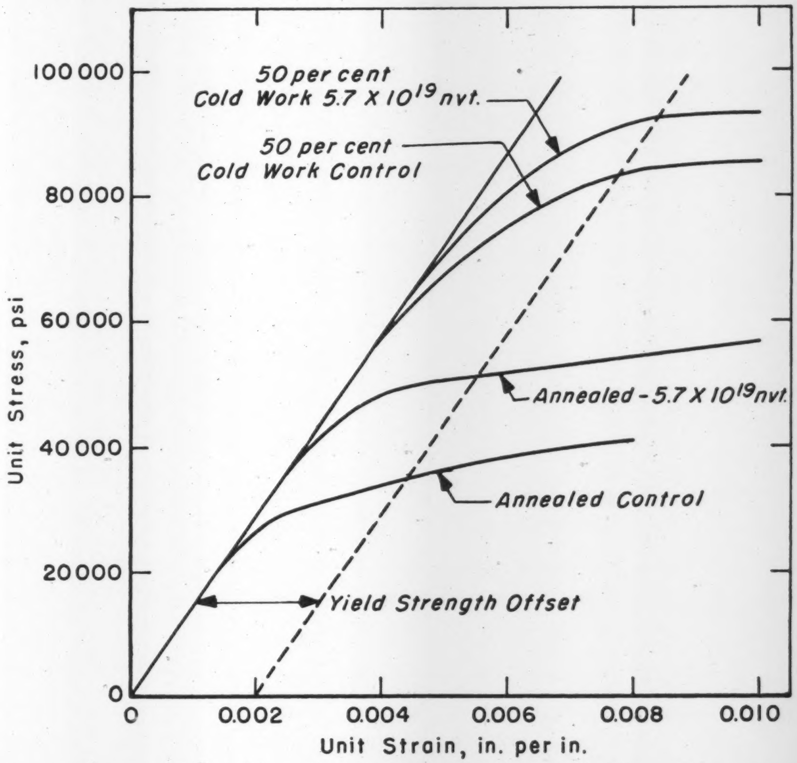

FIG. 2.—Typical Initial Portion of Stress-Strain Curves Illustrating the Increase in Yield Stress Caused by Cold Work and Irradiation.

Strain rate 0.001 in. per in. per min.

These results agree, in general, with hardness data for other types of zirconium[7] and zirconium alloys[8] exposed at approximately the same temperature, although the magnitude of the radiation hardening in both the annealed and cold-worked specimens is greater than that reported for crystal bar zirconium at saturation.[7]

TENSILE PROPERTIES

Typical stress-strain curves for control and irradiated specimens are shown in Fig. 2. The increase in yield stress resulting from cold work or strain hardening is illustrated as well as the increase due to radiation-hardening. No changes were observed in the modulus of elasticity over the error associated with its determination. Figures 3, 4, and 5 show the changes produced in the yield strength, tensile strength, and ductility at each level of cold work for the three exposures. Duplicate samples were tested for the control material and after the first exposure. Excellent reproducibility was obtained in these tests, so at the higher exposures one specimen of each pair was used for post-irradiation annealing.

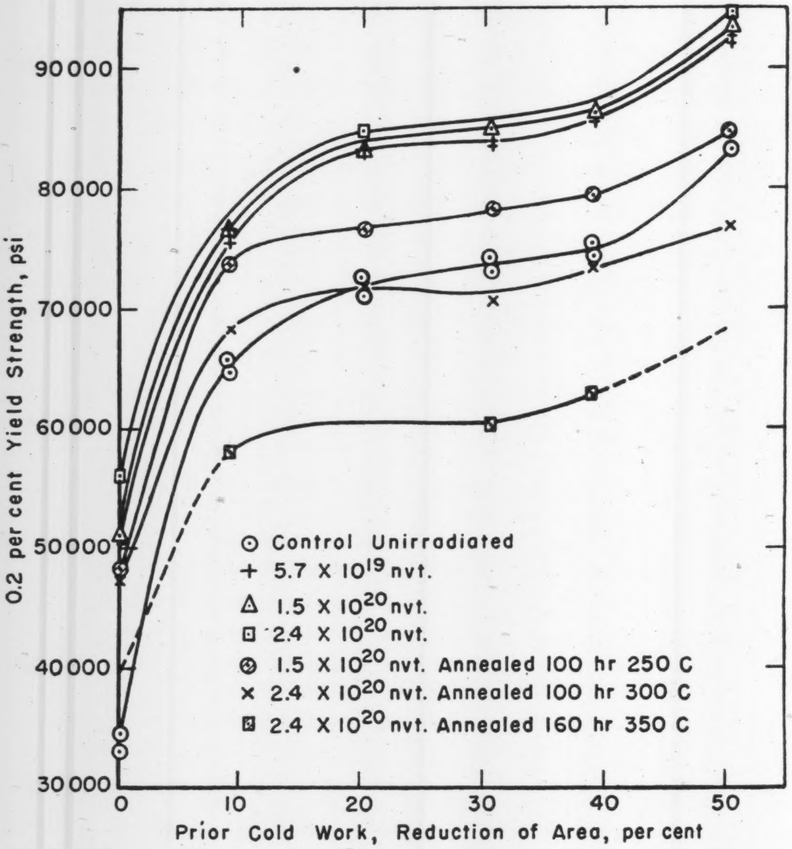

FIG. 3.—Effect of Irradiation and Post-Irradiation Annealing on the Yield Strength of Arc-Melted Bureau of Mines Zirconium.

Exposure Temperature, 50 to 60 C

A detailed discussion of the effects of fast neutron radiation on the mechanical properties of metals has been presented by Dienes.[15] The major result is a displacement effect in which atoms are knocked out of their normal lattice positions leaving vacant lattice sites and coming to rest in interstitial positions. The interaction of the interstitial atoms, or groups of these atoms, with the stress field of dislocations in such a way as to impede the motion of dislocations is felt to be responsible for the hardening and the increase in the stress to produce plastic flow. Holden and Kunz[16] have proposed that during room temperature irradiation, or in warming to room temperature after a low-temperature irradiation, the interstitials produced diffuse and agglomerate to form planar defects which are homologous with stacking faults. Moving a dislocation through such faults should be more difficult than moving it through a random arrangement of individual interstitials and vacancies, resulting in the magnitude of radiation-hardening and increase in flow stress that is observed.

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[15] G. J. Dienes, "Effects of Nuclear Radiations on the Mechanical Properties of Solids," *Journal of Applied Physics*, Vol. 24, No. 6, June, 1953, pp. 666–674.

The blocking of the movement of dislocations by interstitials or faults becomes less pronounced at high strain values where the mobility of dislocations is more affected by their interaction with other dislocations. Thus, the ultimate strength and total ductility are not appreciably altered by radiation.

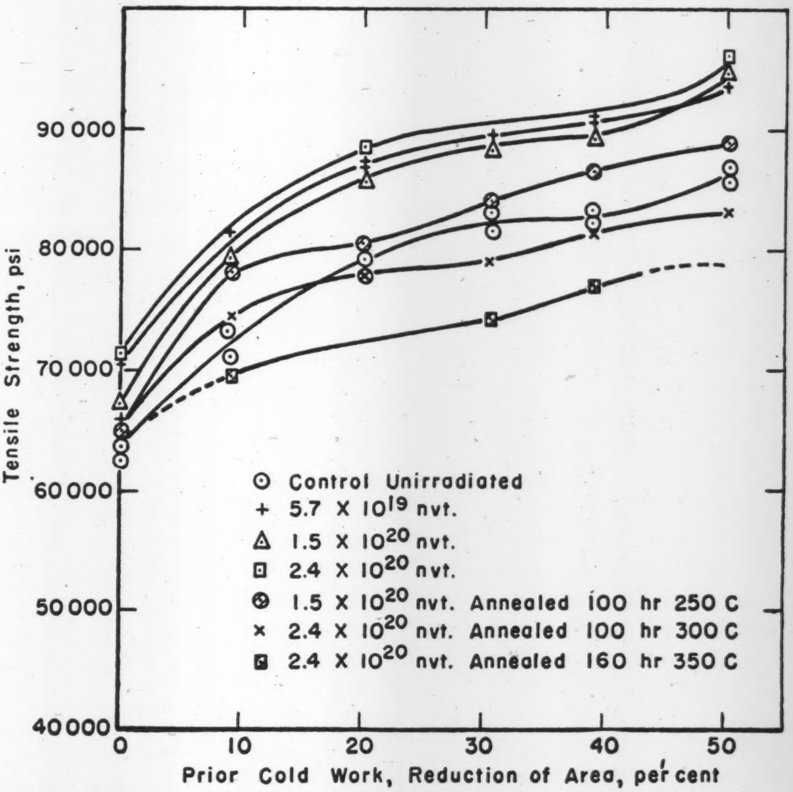

FIG. 4.—Effect of Irradiation and Post-Irradiation Annealing on the Tensile Strength of Arc-Melted Bureau of Mines Zirconium.

Exposure Temperature, 50 to 60 C

The similarity between cold work or strain-hardening and radiation-hardening has been noted although there is no direct correspondence between the two effects. The major differences are that plastic deformation produces organized lattice disturbances from the motion and multiplication of dislocations[15] and that cold-working produces vacancies, but a limited number of interstitials.[17] It is generally observed that after cold work the effect of irradiation is much smaller than in annealed material.[15]

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[16] A. N. Holden and F. W. Kunz, "A Model for Radiation Damaged Ductile Metals," KAPL-929, June 10, 1953 (DECLASSIFIED).

The general effects of neutron irradiation noted in the preceding discussion are evident in the data shown in Figs. 2 through 5. The most pronounced change was observed in the yield strength. The largest increase was noticed in the annealed material in which the change was approximately the same as that produced by cold working to 5 per cent reduction of area. The curves shown in Fig. 6 indicate that the major portion of the damage occurred during the lowest exposure and that for this exposure temperature the damage has essentially saturated.

The increases in tensile strength resulting from the irradiation were more uniform with increasing prior cold work in contrast to the increases in yield strength, which were greater in the annealed material. These changes are shown in Fig. 4. The tensile strength was also not appreciably increased at higher exposures after the initial irradiation.

The ductility, as measured by elongation and reduction of area during the tension test, was decreased slightly by the irradiation. The two curves drawn

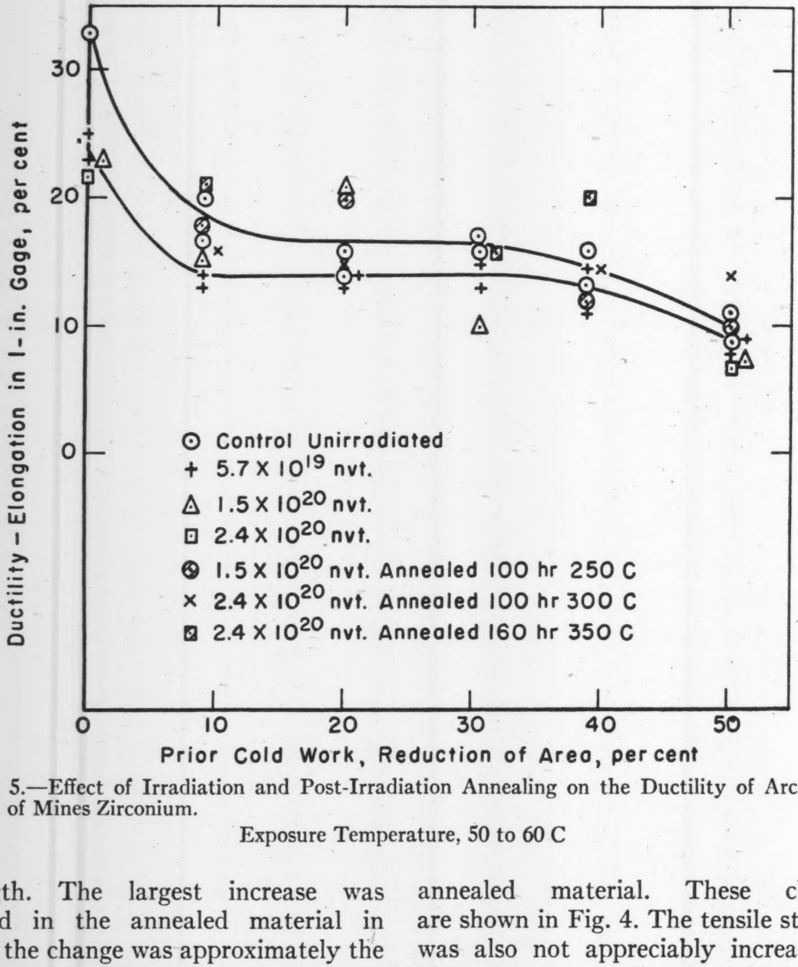

FIG. 5.—Effect of Irradiation and Post-Irradiation Annealing on the Ductility of Arc-Melted Bureau of Mines Zirconium.

Exposure Temperature, 50 to 60 C

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[17] F. Seitz, "Advance in Physics," *Philosophical Magazine*, Supplement 1, 1952, pp. 43–90.

through the data points in Fig. 5 represent the control and initial exposure data. The values for reduction of area are not shown but indicate essentially the same slight decrease in ductility after irradiation.

ANNEALING STUDIES

Post-irradiation annealing of tension specimens was performed at temperatures that would be reasonable for the use of this material in reactor structural components. From the results of long periods of annealing, it was felt that the effect of exposure temperature could be estimated and the recovery of cold work and radiation damage compared. The results of these tests are shown in Figs. 3, 4, and 5. The yield strength is most sensitive to the recovery process and a percentage recovery was defined based on the annealed, pre-irradiation yield strength. Similar material that had been cold worked to approximately 50 per cent reduction of area, but not irradiated, was also annealed for the same period of time at these temperatures for comparison. The percentage recovery of (1) radiation damage only (initially annealed material), (2) radiation damage and cold work, and (3) cold work only are shown in Fig. 7. The data for the 350 C (660 F) anneal are based on 160 hr, and the time at the other temperatures was 100 hr. It is believed that this does not significantly alter the curves or the indication that the recovery of radiation-hardening proceeds at a more rapid rate than the recovery of strain-hardening at temperatures below that

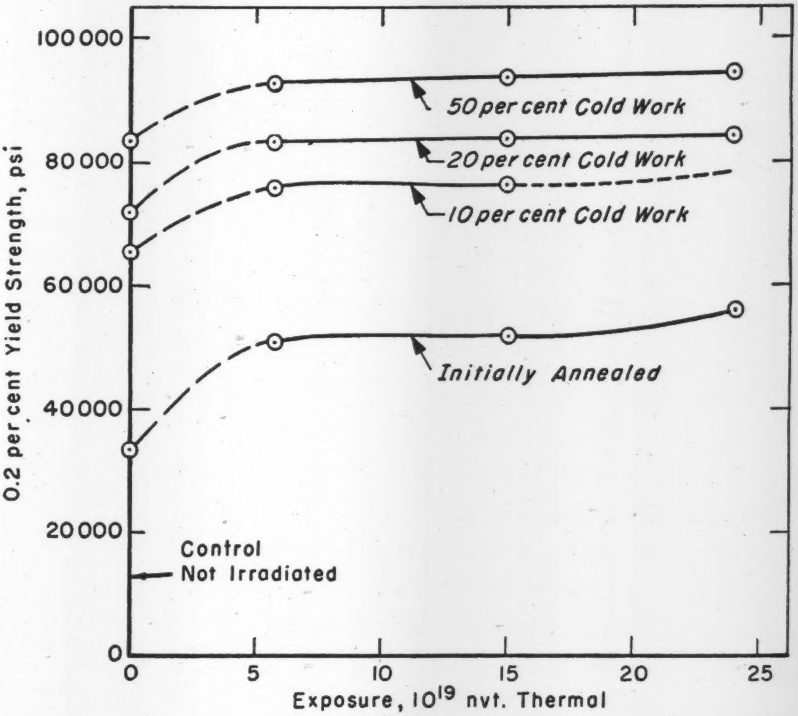

FIG. 6.—Variation of Yield Strength with Exposure Level Illustrating Saturation of Radiation Damage.

Exposure Temperature, 50 to 60 C

required for recrystallization. The only other investigation of recovery of radiation damage to the mechanical properties of zirconium is that of Bleiberg and Castleman[7] who compared the pre- and post-irradiation annealing of hardness in 96 per cent cold-worked crystal bar zirconium at temperatures of 300 to 500 C (575 to 930 F) for 30-min annealing periods. No difference was detected in the hardness decrease of the unirradiated and irradiated, 96 per cent cold-worked material. The radiation-induced hardness in annealed, irradiated crystal bar zirconium was almost completely annealed out after 30 min at 500 C (930 F), and it was concluded that the irradiation-induced hardness anneals out in a manner similar to that for cold-work-induced hardness.

The results of the annealing studies indicate that only slight changes in tensile properties would be incurred during irradiation of annealed zirconium if the exposure temperature were 300 C (575 F) or greater. For cold-worked material irradiated between 300 and 350 C (575 and 660 F), the decrease in yield strength from annealing of the cold work would be expected to more than offset any increase from radiation-hardening.

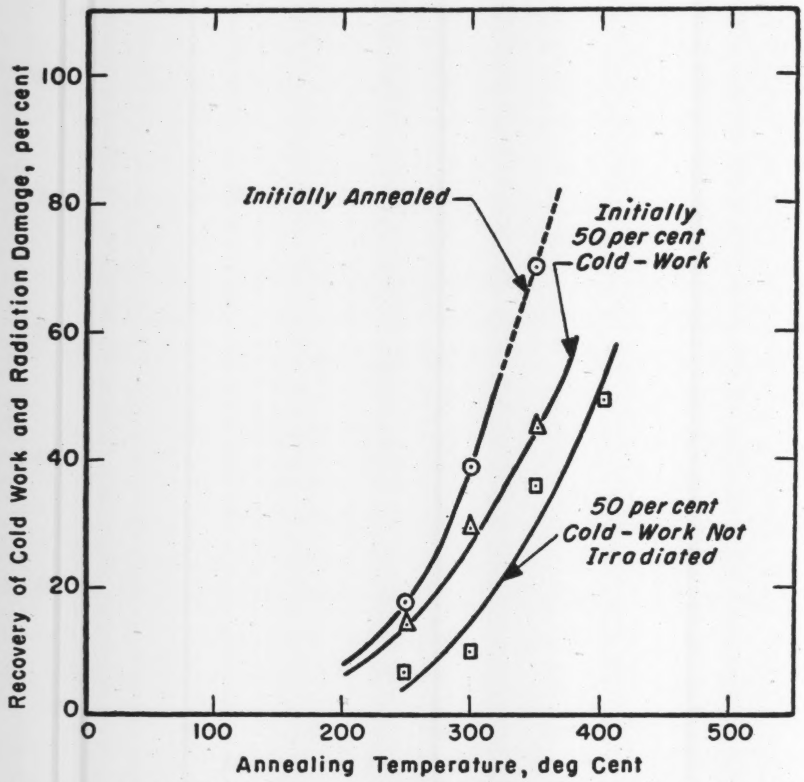

FIG. 7.—Recovery of Radiation Damage and Cold Work during Vacuum Annealing of Irradiated Zirconium.

$$\text{Per cent recovery} = \frac{YS_f - YS_{fa}}{YS_f - YS_b}$$

YS_f = Yield strength, after irradiation and/or cold work
YS_{fa} = Yield strength, after irradiation and/or cold work plus intermediate anneal
YS_b = Yield strength base anneal

CONCLUSIONS

The maximum hardness increase resulting from irradiation was observed in the annealed material, which increased 20 points Rockwell G (50 to 70). The hardness increase was progressively less with increasing prior cold work with the 50 per cent cold-worked material increasing 10 points Rockwell G (65 to 75). The hardness appeared to approach a saturation level at the higher exposures that was essentially independent of prior cold work.

Examination of the microstructure of the irradiated material at 250× did not reveal any changes due to neutron irradiation or thermal conditions during exposure.

The most pronounced effect of irradiation on the tensile properties was noted in the yield strength. A maximum increase of 65 per cent occurred in the annealed material. The change in yield strength was also less for material with prior cold work. In contrast, a more uniform increase in ultimate strength and decrease in ductility resulted, independent of the cold-work level. The change in these properties was not drastic and the material still behaved in a ductile manner. The effect of increased exposure was slight, and further irradiation at this temperature would be expected to produce only small changes in tensile properties over those reported.

Annealing for 100 hr at 250 C (480 F) produced a measurable recovery of the radiation-induced increase in yield strength, and approximately 70 per cent of the damage in initially annealed material was removed in 160 hr at 350 C (660 F). Post-irradiation annealing of the cold-worked material produced recovery of both radiation-hardening and cold work that was dependent upon the degree of prior cold work.

A comparison of the annealing results for irradiated material and unirradiated, 50 per cent cold-worked zirconium revealed greater recovery of radiation-hardening than of strain-hardening for the same temperature and time.

The effects of irradiation observed in this work are in agreement with the effects predicted for interstitial-vacancy pair formation. Saturation of this damage is attributed to attainment of equilibrium between formation and decay of these defects under the conditions of the exposure.

Acknowledgment:

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AN AUTOGRAPHIC BEARING-STRENGTH TEST, AND TYPICAL TEST VALUES ON SOME MAGNESIUM ALLOYS AT ROOM AND ELEVATED TEMPERATURES*

By A. A. Moore[1] and J. A. Gusack[1]

Synopsis

This paper presents a simplified method of measuring hole elongation in the bearing testing of metals and discusses some of the variables and problems encountered in making a bearing test. Bearing properties of some magnesium alloys at room- and elevated-temperatures are presented.

Use of riveted components in aircraft requires that the bearing strength, commonly called the "hole pull-out" strength, be known for the materials used. A bearing yield strength, to define limits of excessive permanent deformation, is also useful. A test using a hardened steel pin to apply the bearing load is used. Several ideas have been advanced on how to define the bearing yield strength (**1**)[2] but the one most commonly used is the nominal stress at the point on the stress-hole-elongation curve which is offset 2 per cent of the hole diameter from the initial straight-line portion of the curve (**2**). The nominal stress is used because the actual stress distribution adjacent to the hole is not uniform (**3**). This stress is obtained by dividing the bearing-pin load in pounds by the projected area of the hole (product of hole diameter and specimen thickness).

Measuring the hole elongation to obtain the bearing yield strength complicated the bearing test about as much as measuring the strain in the tension-test specimen to get the yield strength complicated the tension test. Some testers have been satisfied with dial gages on the head of the testing machine to measure the displacement of the pin relative to the specimen. This method includes such extraneous movements as bending of the pin and tensile strains in the specimen. A more precise method is to determine the movement of the pin by measuring the opening between the pin and the hole boundary with a filar micrometer microscope (**4**). This method is slow and not too suitable for use at elevated temperatures. A third method measures the hole elongation by measuring the displacement of the steel bearing pin relative to the specimen with dial gages (**5**). This method, although accurate, is also slow.

Autographic Bearing Method

Since the accuracy required for a bearing-yield-strength determination should be about the same as for a tensile-yield-strength determination, it was thought that the same strain-measuring equipment could be used for both tests if a suitable transfer mechanism could be devised to translate the hole elongation into a measurable linear movement. Such a transfer mechanism is shown in Fig. 1.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] Metallurgical Laboratory, The Dow Chemical Co., Midland, Mich.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 841.

The principles of operation of the transfer mechanism can be more easily understood by reference to the schematic drawing in Fig. 2. With the specimen in place, the plunger guide is securely positioned on the specimen with the adjustable screws in a relatively low-stressed area. The plunger bears on the pin as close to the specimen as possible to minimize errors due to bending of the pin. Since the displacement of the bearing pin relative to the low-stressed material at the screw locations is the elongation of the bearing hole, the displacement of the plunger relative to the plunger guide is a true measure of the hole elongation. It should be noted that tensile strains in the body of the specimen are not included in this measurement. A Templin autographic extensometer and recorder is used to measure the displacement of the plunger relative to the plunger guide. A curve similar to the load *versus* hole-elongation curve shown in Fig. 3 is obtained by the use of the autographic method.

Tests were made comparing bearing yield strengths determined on the same material with the filar micrometer microscope to measure hole deformation, and the transfer device shown in Fig. 1 with Tuckerman extensometers as the measuring gages. These tests were then compared with bearing yield strengths determined with the same transfer device, but using Templin autographic strain-recording equipment. The bearing yield strengths determined by any of the above methods fell within the same scatter band.

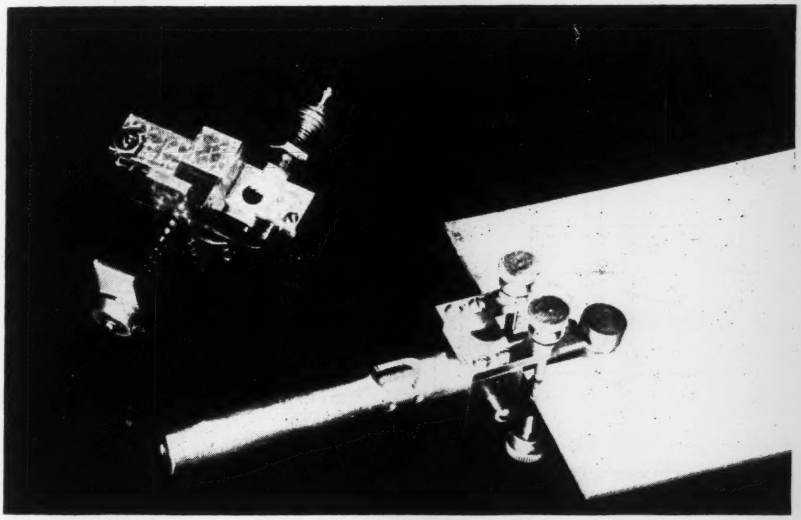
Fig. 1.—Hole-Elongation Transfer Device and Templin Extensometer.

While the use of the strain-transfer device made bearing testing much faster and easier at room temperature, the full advantage was not realized until work at elevated temperatures started. By merely extending the length of the plunger and guide, it is possible, once the temperature is stabilized, to measure the hole elongation outside the heating furnace with no loss in accuracy. Figure 4 shows the transfer mechanism being used in conjunction with standard Templin strain-recorder equipment in making an elevated-temperature bearing test. The electric furnace was lowered for the photograph.

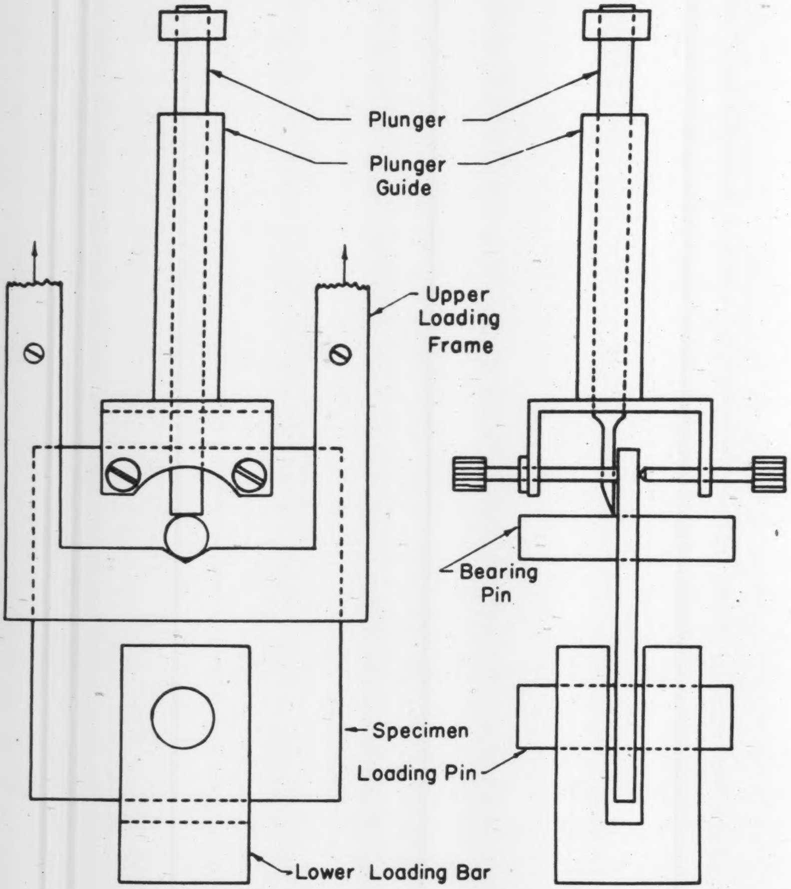

FIG. 2.—Schematic Drawing of Transfer Device Operation.

In elevated-temperature testing, it is important that the temperature be uniform in the bearing specimen adjacent to the bearing pin. To determine the temperature distribution in this area, six thermocouples were buried in a dummy specimen. After the specimen reached thermal equilibrium at 600 F, five of the thermocouples read exactly the same, while the thermocouple directly under the bearing pin read 2 F low. The excellent temperature distribution appears to stem from the use of a long furnace and a loading fixture that allows good circulation of air on the specimen.

For routine testing, it is desirable to eliminate the necessity of applying a new

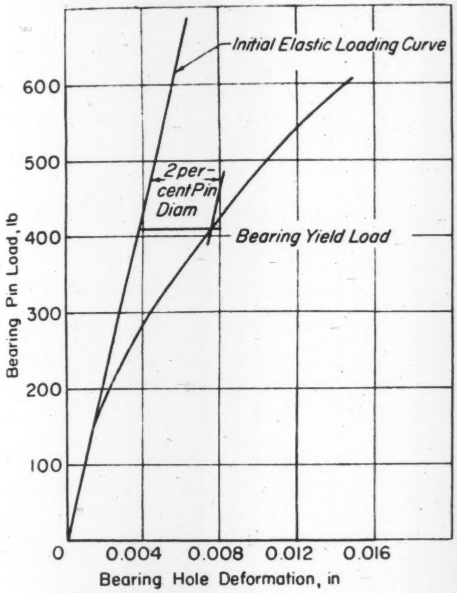

FIG. 3.—An Autographic Load *versus* Hole-Elongation Curve Obtained on AZ31A-H24 Sheet at Room Temperature.

thermocouple to each specimen. To accomplish this, a single thermocouple was silver-soldered to one of the fixed screws in the plunger guide (see Fig. 5). The dummy specimen was allowed to stabilize at 600 F. The thermocouple silver-soldered on the plunger guide read 5 F low compared with the thermocouple buried in the specimen. The thermocouple on the fixed screw is currently being used to monitor the temperature of the specimen, a small correction being applied to the indicated reading at the various testing temperatures.

The temperature of the furnace is controlled by a Micro-Max recorder-controller which receives a signal from a thermocouple mounted close to the heating elements in the furnace. In operation, the recorder-controller is adjusted until the desired specimen temperature is obtained. This method minimizes temperature over-shoot and fluctuation. The current heating time to temperature stabilization is 1 hr.

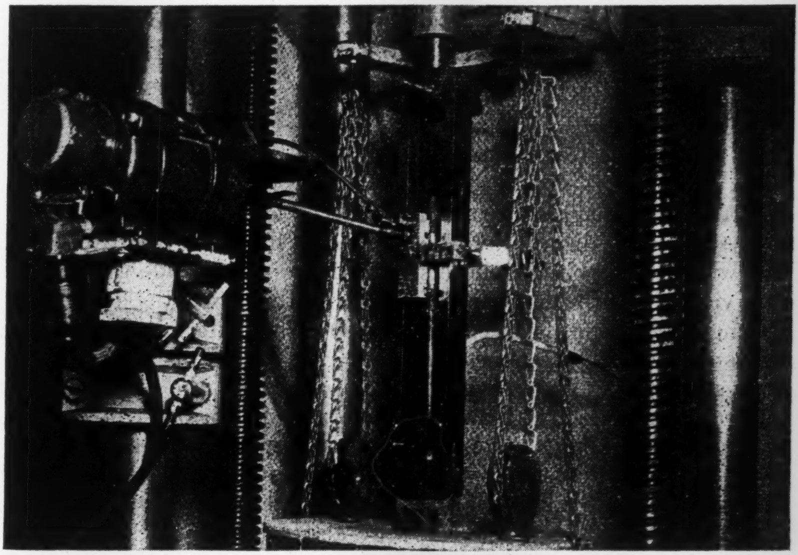

FIG. 4.—Elevated-Temperature Bearing Test Equipment.

BEARING-TEST SPECIMENS

No standard bearing-test specimen has been adopted by ASTM or other national specification-writing agency. Therefore several size factors affecting the results were checked; the most important of these will be discussed.

Different pin sizes do not appreciably change the bearing-yield-strength value obtained on the same magnesium alloy. Decreasing the edge distance ratio, thickness or width of the specimen lowers the bearing yield strength less than the bearing strength. Therefore size effects will be discussed primarily in connection with bearing strength.

If a thin specimen is used with a relatively large pin, a buckling failure will result rather than the "crushing" type of failure generally regarded as bearing failure. For magnesium alloys, a thickness of not less than one-fourth of the pin diameter prevents buckling. Larger pins give lower bearing strengths, probably because the metal at the side of the hole which tends to restrain the movement of the metal above the hole has less effect as the size increases. This effect was also found by R. L. Moore in his work on magnesium alloys using 0.250- and 0.500-in. diameter pins (**6**). No difference could be found on results obtained with 0.187-in. diameter compared with those obtained with 0.250-in. diameter pins. The bearing strengths determined using 0.500-in. diameter pins average about 10 per cent lower on the same material compared to the bearing strengths determined using 0.187-in. diameter pins, with an edge distance of $2\frac{1}{2}$ times the pin diameter.

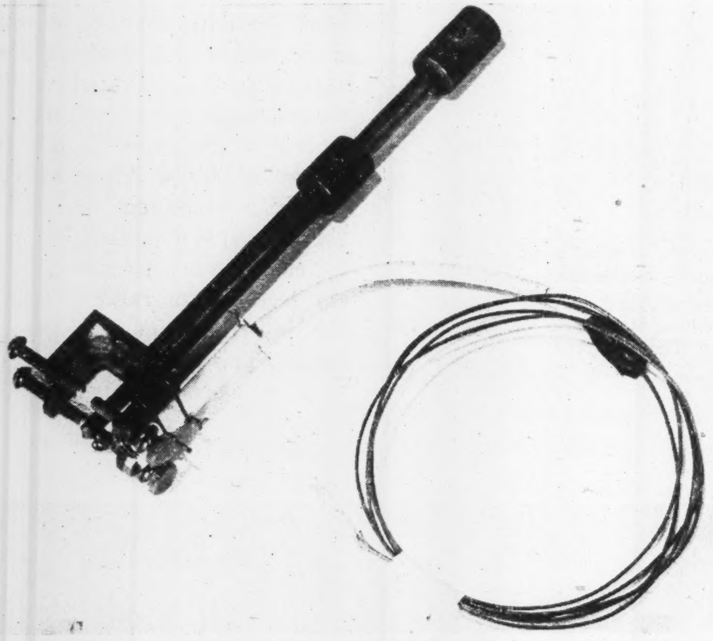

FIG. 5.—Elevated-Temperature Transfer Device and Monitor Thermocouple.

Hole preparation and snugness of pin fit also affect the results obtained. A cold-worked area adjacent to the hole is not desirable because it changes the characteristics of the material being tested. A loose-fitting pin or an out-of-round hole gives reduced bearing strengths. Using a loose-fitting 0.500-in. diameter pin, bearing strengths are lowered by as much as 20 per cent from those obtained with a tight fit. On magnesium alloys, it is found desirable to bore the 0.500-in. diameter holes. The 0.187- and 0.250-in. diameter holes are carefully drilled and reamed.

TABLE I.—TYPICAL BEARING STRENGTHS OF MAGNESIUM ALLOYS AT VARIOUS EDGE DISTANCES.

Alloy, Temper and Form	2.5*D* Edge Distance		2.0*D* Edge Distance		1.5*D* Edge Distance	
	Bearing Yield Strength, psi	Bearing Strength, psi	Bearing Yield Strength, psi	Bearing Strength, psi	Bearing Yield Strength, psi	Bearing Strength, psi
AZ31A-0 sheet	38 100	70 000	...	68 200	38 100	58 100
AZ31A-H24 sheet	53 700	78 500	...	...	...	...
AZ31A-F extrusion	33 200	62 000	33 100	56 200	31 700	45 300
AZ61A-F extrusions	43 600	76 500	41 000	68 100	39 600	57 300
AZ80B-F extrusions	50 400	76 100	48 300	68 200	42 000	57 200
MIA-F extrusions	25 300	53 100	25 400	49 200	24 100	39 600
AZ92A-T4 castings	48 800	77 200	45 700	69 300	40 900	56 700
AZ92A-T6 castings	66 400	90 800	64 600	82 500	53 900	62 800
AZ63A-T4 castings	45 800	71 200	44 000	57 700	36 500	43 800
AZ63A-T6 castings	56 000	83 300	52 600	76 400	44 100	60 500

TABLE II.—MINIMUM DESIGN BEARING PROPERTIES OF MAGNESIUM ALLOYS (**2**).

Alloy, Temper and Forms	1.5*D* Edge Distance		2.0*D* Edge Distance	
	Bearing Yield Strength, psi	Bearing Strength, psi	Bearing Yield Strength, psi	Bearing Strength, psi
AZ31A-0 sheet	29 000	50 000	29 000	60 000
AZ31A-H24 sheet	43 000	58 000	43 000	68 000
AZ31B-F extrusions	23 000	36 000	23 000	45 000
AZ61A-F extrusions	28 000	45 000	32 000	55 000
AZ80A-F extrusions	36 000	48 000	40 000	56 000
MIA-F extrusions	23 000	36 000	23 000	45 000
AZ92A-T4 castings	32 000	48 000	40 000	58 000
AZ92A-T6 castings	45 000	52 000	55 000	70 000
AZ63A-T4 castings	32 000	36 000	36 000	50 000
AZ63A-T6 castings	36 000	50 000	45 000	65 000

The width of the specimen must be sufficient to prevent a tension failure at the hole. The width necessary to prevent tension failure will vary with the ratio of tensile strength to bearing strength and the notch sensitivity of the material. For the tests on magnesium alloys, a specimen width of 8*D* is used, where *D* is the hole diameter. A width of 4*D* is actually satisfactory for most commercial alloys, the wider specimen having been adopted to accommodate occasional special alloys of low ductility.

Various edge distances (measured along the load axis) are used to simulate riveted structure. Edge distances commonly used are 1.5*D*, 2.0*D*, 2.5*D* and 4*D* from the edge of the specimen to the center line of the hole. At an edge distance of 1.5*D*, however, a double-shear

TABLE III.—ELEVATED-TEMPERATURE BEARING PROPERTIES OF THE SAND-CAST MAGNESIUM ALLOY EZ33A-T5.

Test Temperature, deg Fahr	Bearing Yield Strength, psi	Bearing Strength, psi
70	41 500	64 000
400	33 000	52 800
500	31 400	49 800
600	19 350	32 200

NOTE.—2.5*D* Edge Distance; 3/16 in. Pin Diameter.

failure is generally obtained on magnesium alloys, at a lower bearing strength than is obtained when a bearing or "crushing" failure occurs at greater edge distances. The values obtained at an edge distance of 2*D* and over are generally the same. The above generalization does not hold true for all alloys and pin sizes. For example, decreasing the edge distance lowers the bearing strength less on the tests with a 0.500-in. diameter pin than on those with a 0.187-in. diameter pin.

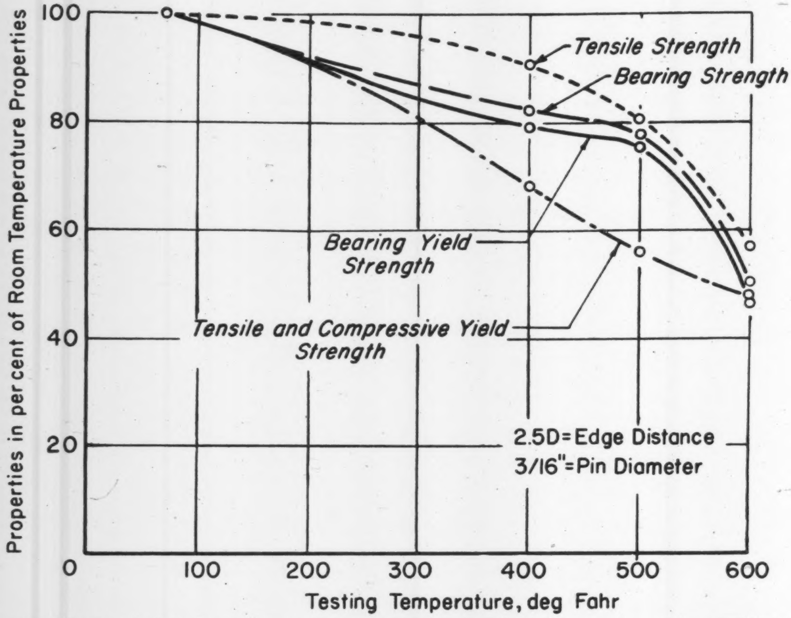

FIG. 6.—Mechanical Property Comparison of the Sand-Cast Magnesium Alloy EZ33A-T5.

At elevated temperatures the standard 8*D* width of specimen is used. An autographic curve is obtained from which the bearing yield strength is readily determined. The bearing-strength determination, however, is complicated by an additional problem not encountered on commercial magnesium alloys at room temperature. At higher temperatures many magnesium alloys become extremely ductile. As the specimen is loaded beyond the yield strength at these temperatures, the metal laterally deforms above the pin, effectively increasing the bearing area. The failure obtained under these conditions is quite different in character from that at room temperature. Because the pin does not pull out until the specimen is severely deformed, errors from specimen interference with the upper loading frame may occur. The material tested at elevated temperatures for this paper did not deform excessively in the temperature range tested, and the results may therefore be considered dependable.

MAGNESIUM-ALLOY BEARING PROPERTIES

Typical room-temperature bearing strengths and bearing yield strengths of some commercial magnesium-base alloys are given in Table I. Enough batches were tested for the values to be considered typical of what can normally be expected from commercial material. The effect of edge distance on the properties can be seen. The minimum bearing strengths and bearing yield strengths for magnesium alloys currently shown in the ANC-5 Bulletin, "Strength of Metal Aircraft Elements" (2), are given in Table II. Elevated-temperature bearing properties of one batch of the sand-cast magnesium alloy, EZ33A-T5, are given in Table III. In general the bearing strength and bearing yield strength decrease with increasing temperature the same as the tensile and compressive properties.

A comparison of the elevated-temperature bearing properties with the elevated-temperature tensile and compressive properties has been made to determine if elevated-temperature bearing properties can be predicted from other routine tests. The results are shown in Fig. 6. No simple correlation was found to relate the routinely determined tensile or compressive properties to the bearing properties. Although the bearing-strength and bearing-yield-strength properties appear to be related for this alloy, it must not be assumed that bearing properties of other magnesium alloys are likewise related. In general, unless further data indicate otherwise, any prediction of bearing properties from tensile or compressive properties of magnesium alloys is only a rough approximation.

CONCLUSIONS

As a result of the work done for this paper, it is concluded that a simplified bearing test method has been established which is little more difficult to perform than a tension test. More work may be needed to determine a suitable specimen for metals other than magnesium. As part of the over-all problem of standardization a bearing test method and definitions of terms should be issued by ASTM, whether or not this property is shown in ASTM material specifications.

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(2) Air Force-Navy-Civil Aircraft Design Criteria Committee, "Strength of Metal Aircraft Elements," *ANC-5 Bulletin*, U. S. Government Printing Office, March, 1955, pp. 131–133, and p. 114.

(3) M. M. Frocht and H. N. Hill, "Stress Concentrations Around a Circular Hole in a Plate Loaded Through a Pin in a Hole," *Journal of Applied Mechanics*, March, 1940, pp. A-5–A-9.

(4) W. H. Sharp and R. L. Moore, "Bearing Tests of Magnesium Alloy Sheet," *Technical Note No.* 897, National Advisory Committee for Aeronautics, June, 1943.

(5) D. E. Miller, "Determination of Physical Properties of Ferrous and Nonferrous Structural Sheet Materials at Elevated Temperatures," *AF Technical Report 6517*, June, 1954.

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EVALUATION OF TEST VARIABLES IN THE DETERMINATION OF SHEAR STRENGTH*

By Raymond W. Fenn, Jr. and Robert B. Clapper[1]

Synopsis

Study of extensive new single shear, double shear, and punch shear test results reveal a significant size effect which must be considered in any attempt to compare shear strengths of alloys. Increased apparent shear strength is observed with: (*a*) decreased specimen shear area, (*b*) increased pin-to-hole tolerance (double shear), (*c*) notching of round specimens, and (*d*) simulated wear of a shear jig. Changes in shear strength as a function of specimen and shear jig geometry are also presented. Recommendations are offered for test methods to provide the best approximation to the true shear strength of the material.

Variations observed in the shear strength of magnesium alloys as determined by the double shear method raised various questions concerning the test variables and what effect they have on the apparent shear strength. What methods, other than the torsion method, can be used to determine ultimate shear strength? What are the true shear strengths of magnesium alloys?

It was the object of this investigation to answer questions such as those given above and to compare test results obtained by the various methods.

At present there is no ASTM standard method for the determination of ultimate shear strength. However, many laboratories use double shear jigs made to a military specification[2] which describes the geometry of a jig for shear testing rivet material. In 1953, F. Gatto[3] proposed a single shear method (see Fig. 1) which was claimed to give pure shear, and consequently higher shear strengths, than those obtained by the double shear method.

Design shear strength values for sheet have previously been obtained by the punch shear method. A method for testing sheet in single shear by Davis, Troxell, and Wiskocil,[4] has been used as a test which more truly represents conditions such as those found in shear-resistant webs of sheet.

The results of a systematic variation of the test variables for the pin double shear method, pin single shear, sheet single shear, and punch shear methods are presented and compared.

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* **Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.**

[1] The Dow Chemical Co., Metallurgical Lab., Midland, Mich.

[2] Military Specification: Rivets; Aluminum and Aluminum Alloy, Aircraft; Mil-R-5674A, *Amendment*—1, Feb. 12, 1953.

[3] F. Gatto, "Shear Test of Light Alloys" (in Italian), *Aluminio*, *22*, 495–506 (1953).

[4] H. E. Davis, G. E. Troxell and C. T. Wiskocil, "The Testing and Inspection of Engineering Materials," McGraw-Hill Book Co., Inc., Eighth printing of preliminary edition, p. 113 (1949).

TEST MATERIALS

The composition and mechanical properties of the alloys are shown in Table I. The majority of double and single shear tests were conducted on pins machined from $1\frac{1}{8}$ in. and 1-in. diameter extrusions of AZ80A-F magnesium alloy, designated as lots I and II, respectively. Supplementary double shear data were drawn from preliminary test results on $\frac{1}{2}$-in. diameter AZ21-F[5] extrusions; mechanical property data were not obtained on this alloy. The materials selected for the single shear sheet and the punch shear programs were AZ31A-O $\frac{1}{2}$-in. plate, 0.064-in. 2024-T3 clad and 7075-T6 bare aluminum sheet. The chemical composition of the 7075 aluminum alloy was not obtained.

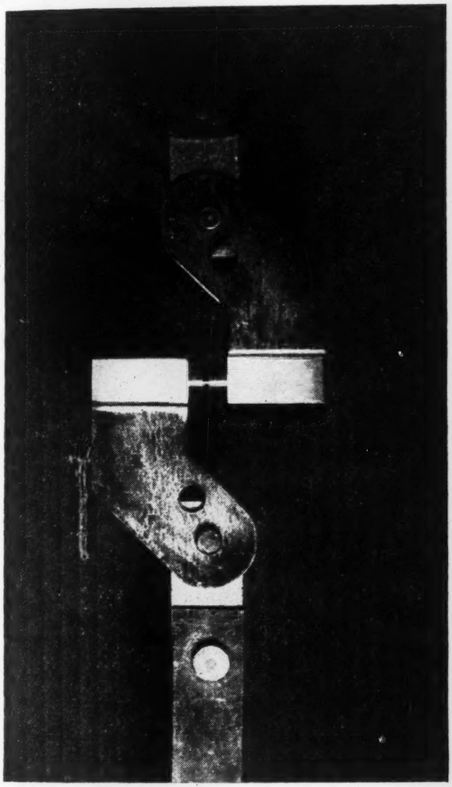

FIG. 1.—Apparatus for Single Shear Testing of Round Specimens.

DOUBLE SHEAR

Apparatus and Procedure:

Double shear tests were made with four different jigs, all of which were based on the principle of the jigs used for testing rivet material.[2] A center shearing plate with several short transverse holes of different sizes is placed between two outer plates having a similar series of holes in such a manner as to align the hole to be used in all three plates. The two outer plates are bolted to a spacer plate of the same thickness as the center shearing plate. This spacer plate serves to align the shearing holes of the two outer plates and extends beyond the end of the jig enough to pass a clevis pin through a hole for the purpose of applying load. The center shearing plate also extends beyond the other ends of the outer shearing plates enough to pass a clevis pin through a hole. In this manner the shear stress is transmitted essentially equally to the two shear planes which are the adjacent planes of the center and outer plates. The bolted arrangement also minimizes (*a*) distortion due to eccentricities, and (*b*) the tendency for the specimen to bend as it tries to spring the outer plates apart. Three jigs were made with center plates $\frac{1}{2}$, 1 and $1\frac{1}{2}$ in. thick and outside plates $1\frac{1}{4}$ in. thick, each having holes for shearing pins of $\frac{1}{8}$ to 1 in. diameter. The other jigs were originally made to a military specification[2] which specifies a different jig for each pin diameter.

The different tests included studies of varying the clearance between shear faces, varying the clearances between the hole and the specimen diameter, and chamfering the edges of holes at the

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[5] AZ21-F is an experimental alloy and the ASTM nomenclature is used simply for convenience.

TABLE I.—MECHANICAL PROPERTIES AND COMPOSITION OF ALLOYS.

Alloy	Material Description	Mechanical Properties					Chemical Composition, per cent					
		Direction of Test	Elongation in 2 in., per cent	Tensile Yield Strength, psi	Tensile Strength, psi	Compressive Yield Strength, psi	Aluminum	Zinc	Manganese	Calcium	Copper	Magnesium
AZ80A-F (Lot I)	1⅛ in. diam. extrusion	Long.	13 Avg.	37 000 Avg.	50 000 Avg.	28 000 Avg.	8.2	0.34	0.20	<0.01	0.003	Bal.[a]
AZ80A-F (lot II)	1.0 in. diam. extrusion	Long.	13 Avg.	37 000 Avg.	50 000 Avg.	30 000 Avg.	7.8	0.43	0.43	<0.01	0.003	Bal.[a]
AZ21-F	½ in. diam. extrusion	Long.	...	...	...	...	1.7	0.50	0.04	0.17	...	Bal.[a]
AZ31A-O	½ in. plate	Long.	21	22 000	36 000	10 000	2.5	0.88	0.56	0.27	0.003	Bal.[a]
AZ31A-O	½ in. plate	Transv.	16	21 000	36 000	11 000	2.5	0.88	0.56	0.27	0.003	Bal.[a]
2024-T3 clad	0.064 in. sheet	Long.	19	50 000	65 000	50 000	Bal.[a]	0.10	0.54	...	4.0	1.9
7075-T6 bare[b]	0.064 in. sheet	Long.	11	77 000	82 000	75 000	Bal.[a]	5.6	Cr = 0.3		1.6	2.5

[a] Balance includes impurities.

[b] Chemical composition for 7075 is nominal.

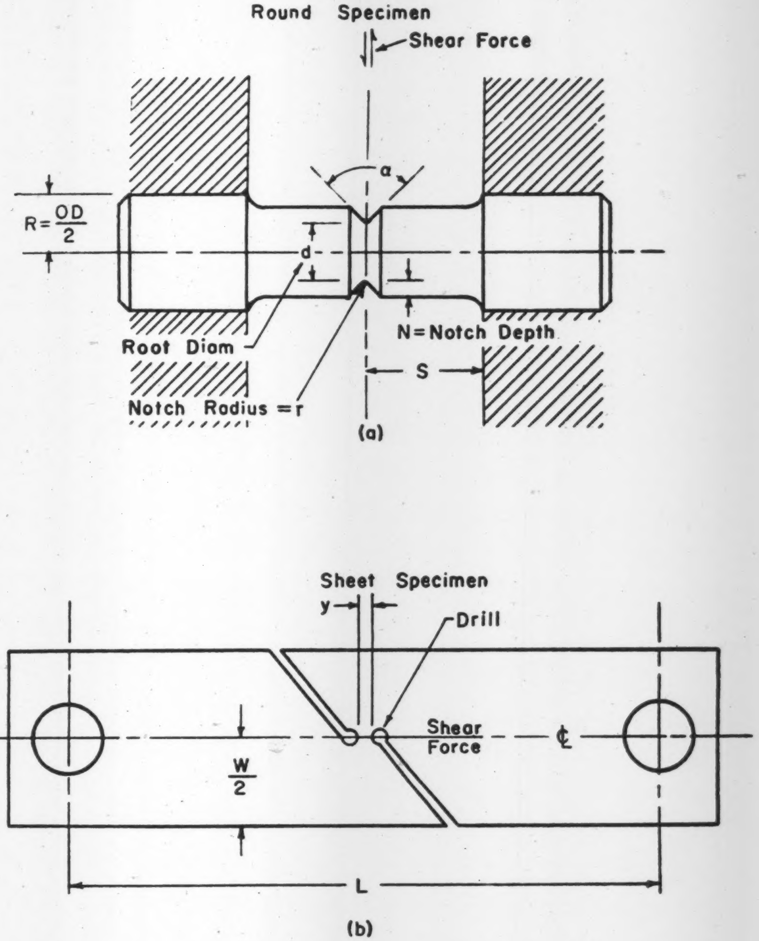
Round Specimen
Shear Force
a
R = OD/2
d
Root Diam
N = Notch Depth
S
Notch Radius = r
(a)
Sheet Specimen
y
Drill
Shear Force
℄
W/2
L
(b)

FIG. 2.—Single Shear Round and Sheet Specimens.

shear faces. The clearance between shear faces was varied by placing shims between the spacer and outer plates under the bolts. Specimen to hole clearance was changed by reducing the specimen diameters. Chamfering the edges of the shear pin holes was done by machining close-fitting bushings which were subsequently inserted in a shear jig.

Notched bar double shear specimens were made similar to that shown in Fig. 2(*a*) except that each specimen was notched in two places to coincide with the shear planes. The undercuts served only to increase the s[6] distance to control the $\frac{s}{R}$ ratio.

TESTS AND RESULTS

A program for the double shear method was designed to determine the

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[6] See Appendix for definition of symbols.

TABLE II.—DOUBLE SHEAR TEST RESULTS ON AZ80A-F MAGNESIUM EXTRUSION (LOT I).

d, in.	Shear Stress, 1000 psi				T, in.	t/d	t, in.
				Avg.			
Group IA—t = 0.498 in.							
0.125	24.1	23.4	23.6	23.7	0.003	3.98	
0.250	22.5	22.7	22.4	22.5	0.002	1.98	
0.375	21.8	22.4	...	22.1	0.004	1.32	
0.500	21.8	22.2	22.0	22.0	0.003	0.98	
0.750	21.8	22.1	22.0	22.0	0.002	0.66	
1.000	20.7	21.0	21.4	21.0	0.003	0.50	
Group IB—t = 0.996 in.							
0.125	24.1	23.5	23.7	23.7	0.003	7.96	
0.250	22.9	22.7	...	22.8	0.001	3.97	
0.375	21.9	22.0	22.0	22.0	0.002	2.65	
0.500	21.6	21.8	22.3	21.9	0.003	1.99	
0.750	21.8	21.7	21.9	21.8	0.002	1.33	
1.000	21.4	21.0	21.6	21.3	0.004	0.99	
Group IC—t = 1.473 in.							
0.125	23.8	24.2	24.4	24.1	0.002	11.8	
0.250	23.2	22.9	23.1	23.1	0.001	5.9	
0.375	21.5	21.8	21.8	21.7	0.001	3.93	
0.500	22.5	22.4	22.3	22.4	0.002	2.94	
0.750	22.4	22.3	22.5	22.4	0.001	1.96	
1.000	21.6	22.1	22.1	21.9	0.002	1.47	
Group ID—t = variable							
0.125	22.4	23.7	22.3	22.8	0.002	1.0	0.126
0.188	22.6	21.8	21.8	22.1	0.002	1.3	0.249
0.188	22.0	22.6	22.7	22.4	0.003	1.3	0.249
0.250	21.7	21.3	21.4	21.5	0.002	0.92	0.231
0.250	22.3	21.8	22.1	22.1	0.002	0.92	0.231
0.375	21.6	22.8	22.8	22.4	0.002	0.96	0.373

S, in.	Shear Stress, 1000 psi			T, in.	t/d
			Avg.		
Group IIA—d = 0.250 in.; t = 1.473 in.					
	22.4	23.8	23.1	0.001	5.9
	22.3	23.1	22.7	0.002	5.9
	23.2	23.2	23.2	0.003	5.9
	23.3	23.4	23.4	0.004	5.9
	23.4	23.3	23.4	0.005	5.9
	23.6	23.5	23.6	0.006	5.9
	23.4	23.2	23.3	0.006	5.9
	23.6	23.5	23.6	0.025	5.9
	23.4	22.4	22.9	0.039	5.9
	23.1	23.0	23.0	0.049	5.9
Group IIB—d = 0.750 in.; t = 1.473 in.					
	22.4	22.1	22.2	0.004	1.96
	22.6	22.4	22.5	0.007	1.96
	22.1	22.6	22.4	0.010	1.96
	22.2	22.6	22.4	0.013	1.96
	22.5	22.5	22.5	0.016	1.96
	22.5	22.5	22.5	0.019	1.96
	22.4	22.5	22.4	0.022	1.96
	21.0	21.2	21.1	0.076	1.96
	20.8	20.7	20.8	0.086	1.96
	20.0	20.1	20.0	0.150	1.96
Group IIIA—d = 0.250 in.; t = 1.473 in.					
0.000	22.8	23.1	23.0	0.002	5.9
0.001	23.3	23.4	23.4	0.002	5.9
0.002	23.4	23.3	23.4	0.002	5.9
0.003	23.4	23.5	23.4	0.002	5.9
0.004	23.4	23.4	23.4	0.002	5.9
0.006	23.4	23.3	23.4	0.002	5.9
0.010	23.2	23.3	23.2	0.002	5.9
0.040	22.7	22.8	22.8	0.001	5.9
0.064	23.0	22.9	23.0	0.001	5.9
0.150	22.6	22.5	22.6	0.002	5.9
Group IIIB—d = 0.750 in.; t = 1.473 in.					
0.000	22.4	22.5	22.4	0.001	1.96
0.001	22.2	22.1	22.2	0.002	1.96
0.002	22.4	22.2	22.3	0.002	1.96
0.003	22.3	22.4	22.4	0.003	1.96
0.004	22.0	22.3	22.1	0.002	1.96
0.006	22.0	22.0	22.0	0.003	1.96
0.010	22.3	22.2	22.2	0.002	1.96
0.040	22.2	22.2	22.2	0.002	1.96
0.062	21.9	21.9	21.9	0.004	1.96
0.107	21.7	21.4	21.6	0.004	1.96

shear strength of AZ80A-F alloy as a function of variations in: (*a*) pin diameter, (*b*) center shear plate thickness, (*c*) pin to hole tolerance, and (*d*) clearance between the center shearing plate and the outside shearing plates, because significant variations in shear strength were obtained for various pin diameters when using double shear jigs made up in accordance with the military specifications.[2] The results of these experiments on plain round bars are tabulated in Table II.

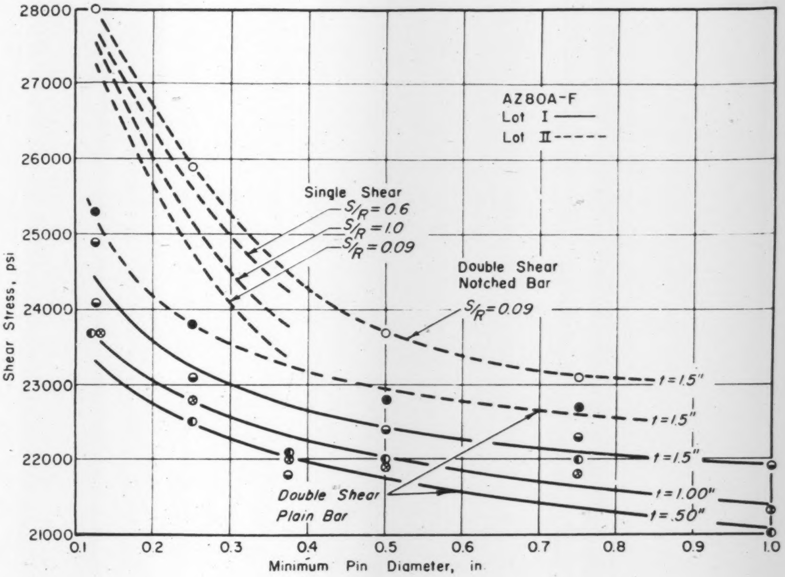

FIG. 3.—Shear Stress as a Function of Minimum Pin Diameter for Varying t and $\frac{s}{R}$.

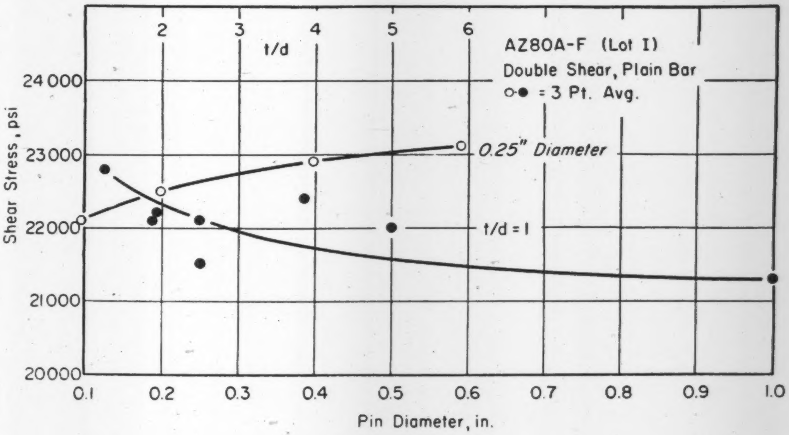

FIG. 4.—Shear Stress as a Function of Pin Diameter for $\frac{t}{d} = 1$ and Varying $\frac{t}{d}$ for $d = \frac{1}{4}$ in.

Groups IA, IB, and IC show the

effect of varying pin diameter with 0.5 in., 1 in., and 1.5 in. thick center shearing plates. The lower three curves of Fig. 3 show shear stress increasing with decreasing pin diameter. A similar size effect was noted when square pins were sheared in shear jigs containing square holes. The lower curve of Fig. 4 shows the increase in shear stress obtained when the ratio of center shear plate thickness to pin diameter $\left(\frac{t}{d}\right)$ is held to approximately one. See Table II, group ID. The upper curve of Fig. 4 shows the gradual increase in shear stress obtained on $\frac{1}{4}$-in. diameter pins with increasing $\frac{t}{d}$. The increases in shear stress observed with increased shear plate thickness are attributed to minimizing the interaction of the two stress systems operating on the two shear planes.

The data of group II A and II B (Table II) show the effect of increasing the pin-to-hole tolerance for $\frac{1}{4}$-in. and $\frac{3}{4}$-in. diameter pins. When the changes in shear stress resulting from the variation in pin-to-hole tolerance are calculated, the data can be combined with those obtained on AZ21-F alloy to give the family of curves for various pin diameters shown in Fig. 5. It is noted that a maximum shear stress is obtained when the pin-to-hole tolerance is between 2 and 4 per cent of the pin diameter, where the 2 per cent applies to the $\frac{3}{4}$-in. diameter pins and the 4 per cent to the $\frac{1}{8}$-in. diameter pins.

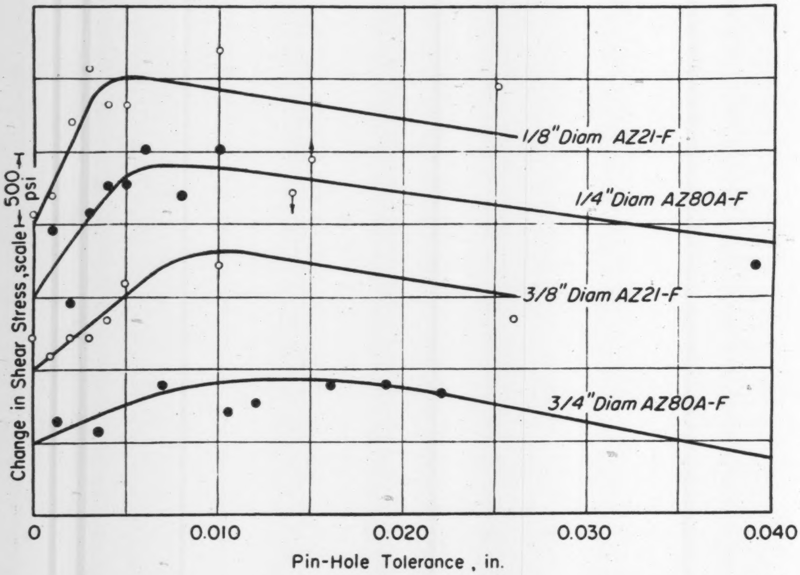

FIG. 5.—Shear Stress as a Function of the Difference in Specimen Diameter and Hole Diameter.

The data of group III A and III B (Table II) show the effect of increasing the total clearance between the center shearing plate and the side plates on the shear stress of $\frac{1}{4}$-in. and $\frac{3}{4}$-in. diameter pins. An increase of approximately 450 psi is noted for the $\frac{1}{4}$-in. diameter pins when the distance between the outside

TABLE III.—DOUBLE SHEAR TEST RESULTS ON AZ80A-F MAGNESIUM EXTRUSION (LOT II).

GROUP IV—UNNOTCHED, 45 DEG CHAMFER, t = 1.473 IN.

Max. diam of pin, in.	C, in.	$C \times 0.707 \times 4$, in.	Shear Stress, 1000 psi				T, in.
						Avg., psi	
0.385	0	0	23.0	23.1	23.9	23.3	0.003
0.385	0.006	0.017	23.4	24.5	23.7	23.9	0.002
0.385	0.016	0.045	26.1	25.0	25.1	25.4	0.003
0.385	0.018	0.051	25.9	26.9	25.7	26.2	0.002
0.385	0.021	0.059	25.4	26.7	25.5	25.9	0.003
0.385	0.028	0.079	25.6	24.1	24.6	24.8	0.003
0.385	0.046	0.130	25.1	25.2	...	25.2	0.002
0.385	0.094	0.266	25.0	24.3	...	24.6	0.003

GROUP V—30 DEG NOTCH, 0.010 IN. ROOT RADIUS, t = 1.473 IN.

Root Diam, in.	Undercut, in.	s, in,	Max. diam of pin, in.	$\frac{s}{R}$ in.	N, in.	Shear Stress, 1000 psi				T, in.	t/d
									Avg, psi		
0.125	0.075 × 0.250	0.125	0.750	0.33	0.312	27.5	27.4	28.0	27.6	0.002	11.8
0.250	0.075 × 0.500	0.250	0.750	0.66	0.250	25.6	26.0	26.1	25.9	0.002	5.9
0.375	0.075 × 0.750	0.375	0.750	1.00	0.188	23.5	23.1	23.9	23.5	0.002	3.9
0.500	0.075 × 1.00	0.500	0.750	1.33	0.125	Specimens Bent				...	2.9
0.125	0.075 × 0.750	0.375	0.750	1.0	0.312	26.7	...	...	26.7	0.002	11.8
0.200	0.075 × 1.126	0.563	0.750	1.5	0.275	26.1	...	...	26.1	0.002	7.4
0.250	0.075 × 0.188	0.094	0.750	0.25	0.250	24.8	24.4	...	24.6	0.002	5.9
0.250	0.075 × 0.750	0.375	0.750	1.0	0.250	25.9	25.8	...	25.8	0.002	5.9
0.250	0.075 × 1.124	0.562	0.750	1.5	0.25	25.8	25.4	...	25.6	0.002	5.9

GROUP VI—30 DEG NOTCH, 0.010 IN. ROOT RADIUS, t = 1.473 IN.

0.125	None	0.084	0.750	0.224	0.312	26.9	26.6	...	26.8	0.002	11.9
0.250	None	0.067	0.750	0.179	0.250	25.0	25.3	25.1	25.1	0.002	5.9
0.375	None	0.050	0.750	0.134	0.187	24.0	23.7	23.6	23.8	0.002	3.9
0.500	None	0.034	0.750	0.089	0.125	23.9	24.0	23.8	23.9	0.002	2.9
0.375	None	0.017	0.500	0.068	0.062	25.6	25.6	25.5	25.6	...	2.0

GROUP VII—30 DEG NOTCH, 0.010 IN. ROOT RADIUS, t = 1.473 IN.

0.125	None	0.034	0.375	0.089	0.125	27.7	28.2	28.0	28.0	0.001	11.9
0.250	None	0.034	0.500	0.089	0.125	26.2	25.6	25.9	25.9	0.002	5.9
0.500	None	0.034	0.750	0.089	0.125	23.1	23.7	23.8	23.5	0.002	2.9
0.750	None	0.034	1.000	0.089	0.125	22.6	23.5	23.1	23.1	0.001	2.0

GROUP VIII—UNNOTCHED CONTROL SPECIMENS, t = 1.473 IN.

0.125	None	0	0.125	0	0	25.0	25.4	25.4	25.3	0.004	11.9
0.250	None	0	0.250	0	0	23.6	24.0	...	23.8	0.002	5.9
0.375	None	0	0.375	0	0	22.2	22.2	...	22.2	0.002	3.9
0.500	None	0	0.500	0	0	22.5	23.1	...	22.8	0.002	2.9
0.750	None	0	0.750	0	0	22.8	22.6	...	22.7	0.000	2.0

shearing plates was increased by 0.003 in.; however, this effect was not observed for the ¾-in. diameter pins. Except for the above increase in shear stress, a gradual decrease is noted with increasing clearance for both pin diameters (Fig. 6). The improved precision of the results of groups IIIA and IIIB over those of the other groups in Table II is attributed to the smaller pin-to-hole tolerances in the former. The pins in groups IIIA and IIIB had to be driven into the jig with a light mallet, whereas those of the other groups were a relatively loose fit.

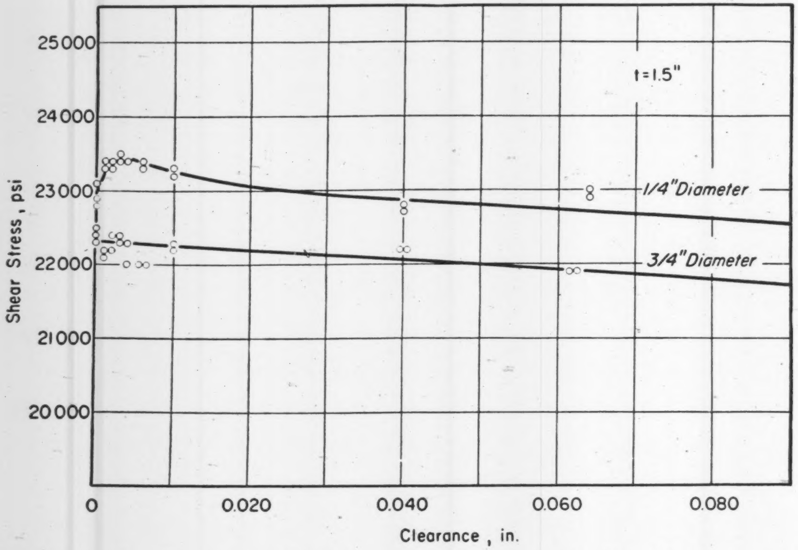

FIG. 6.—Shear Stress as a Function of the Clearance between the Shearing Plate and the Side Plates of the Double Shear Jig.

The shear stress results of a second set of double shear experiments, designed to show the effect of (*a*) chamfering the shearing edges of a shear jig, (*b*) notching specimens with an undercut area, (*c*) notching specimens without an undercut area, and (*d*) notching specimens to a constant depth, are presented in Table III. A plot of the group IV results (Fig. 7) shows an increase in shear stress of approximately 140 psi per 0.001-in. width of 45 deg chamfer face (on each of the four shearing faces) up to a maximum of 0.030 in. Above 0.030 in., increases in width of the chamfered faces result in a gradual decrease in shear stress. Application of the correlation analysis of Ezekiel[7] to the results of groups V, VI, VII, and VIII show: (*a*) that variations in the ratio of the distance from the shear plane to the point of specimen support (s) to one half the maximum specimen diameter (R) cause significant variations in shear stress similar to those shown in Fig. 8, and (*b*) that notch depth has no apparent effect. In the interests of brevity this analysis is not included and the double shear data points of groups V, VI, VII, and VIII are shown in Fig. 8. The effect of varying pin diameter with constant notch depth and constant ratio of $\frac{s}{R}$ (0.09) (group V data) is demonstrated in the top curve of Fig. 3. The notching of the double shear

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[7] M. Ezekiel, "Methods of Correlation Analysis," John Wiley and Sons, Inc., New York, second edition, chap. 16 (1950).

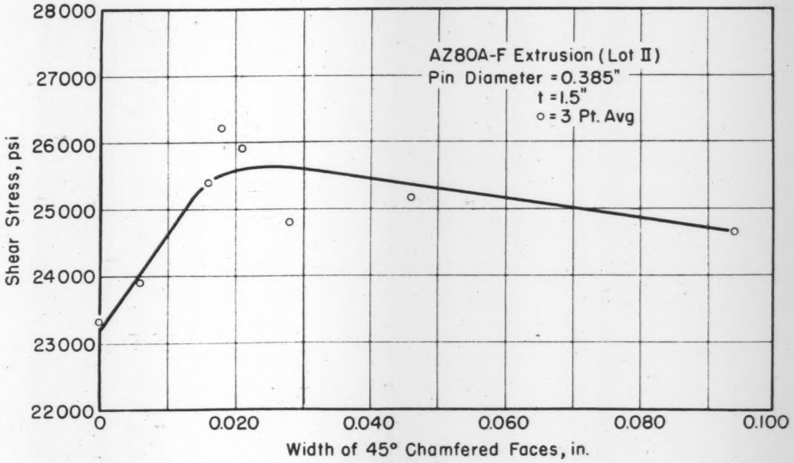

FIG. 7.—Shear Stress as a Function of Chamfering the Edges of the Double Shear Jig Holes.

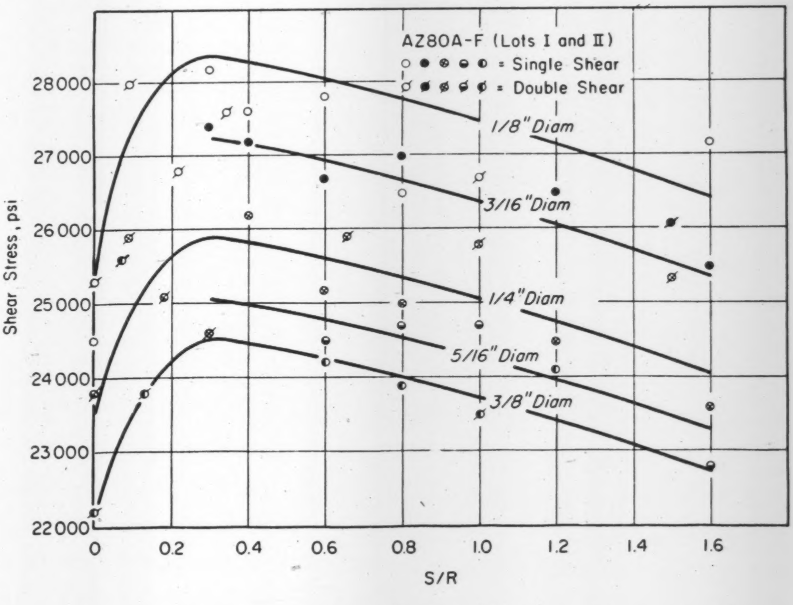

FIG. 8.—Shear Stress as a Function of $\frac{s}{R}$ Values for Various Diameter Specimens.

TABLE IV.—SINGLE SHEAR TEST RESULTS FOR LOTS I AND II AZ80A-F EXTRUSIONS, WITH VARYING PIN DIAMETER AND SUPPORT DISTANCE.

d, in.	N, in.	Shear Stress, 1000 psi α = 30 deg, r = 0.010 in.								
		Lot I	Lot II							
		s = 0.500 in. s/R = 1.600	s = 0.062 in. s/R = 0.200	s = 0.094 in. s/R = 0.300	s = 0.125 in. s/R = 0.400	s = 0.188 in. s/R = 0.600	s = 0.250 in. s/R = 0.800	s = 0.313 in. s/R = 1.000	s = 0.375 in. s/R = 1.200	s = 0.500 in. s/R = 1.600
0.125	0.237	27.0 27.5 25.7 28.6 27.2 Avg.	25.2 23.5 25.0 24.6 Avg.	27.8 28.6 28.1 28.2 Avg.	27.1 27.3 28.5 27.6 Avg.	27.8 27.9 27.7 27.8 Avg.	26.1 27.1 26.3 26.5 Avg.			
0.188	0.269	25.8 25.5 25.1 25.5 Avg.		27.2 26.9 28.0 27.4 Avg.	26.9 26.7 28.1 27.2 Avg.	26.8 26.7 26.6 26.7 Avg.	27.2 26.9 27.0 27.0 Avg.		26.7 26.6 26.3 26.5 Avg.	
0.250	0.179	23.4 23.9 24.1 23.1 23.6 Avg.			27.6 25.8 25.2 26.2 Avg.	25.0 25.2 25.5 25.2 Avg.	24.9 24.1 25.9 25.0 Avg.		24.8 24.2 24.4 24.5 Avg.	24.1 25.1 25.8 25.0 Avg.
0.313	0.207	23.0 21.6 23.3 23.3 22.8 Avg.				24.8 23.8 24.9 24.5 Avg.	24.3 24.7 25.0 24.7 Avg.	24.6 24.8 24.7 24.7 Avg.		
0.375	0.239					24.6 24.0 24.3 24.3 Avg.	23.9 24.0 23.7 23.9 Avg.		24.5 24.0 23.9 24.1 Avg.	

specimens causes even larger increases in shear stress for the smaller diameter pins than observed with plain unnotched specimens. This is attributed to the increase in $\frac{s}{R}$ ratio. The control data for AZ80A-F lot *II* (group VIII) shows the same diameter effect as noted in lot *I*, however the shear stress of the second lot of AZ80A-F is approximately 600 psi higher than the first lot.

SINGLE SHEAR OF NOTCHED ROUND SPECIMENS

Apparatus and Procedure:

Single shear testing was done on the apparatus shown in Fig. 1. Considerable care was taken to be certain the alignment of the root of the notch was exactly in the shear plane, or centerline of shear force as shown in Fig. 2(*a*). The jig, clevis pins, and specimen were adjusted and aligned visually with the aid of: (*a*) two sight glasses containing holes spaced to fit exactly over the two clevis pins, and (*b*) lines scribed on the glass between the centerlines of these holes. The root of the notch was then shown clearly with respect to alignment with the scribed lines, and the specimen was secured against movement by tightening set screws in the jig which clamped the specimen firmly in place.

TESTS AND RESULTS

Gatto's work with single shear apparatus on round specimens indicates that a pure shear condition results at the shear plane of a specimen tested as shown in Fig. 1. He found that a notch at the shear plane insured failure in the specimen at this point, otherwise a bending failure would result from the bending moment which increases from zero at the shear plane to a maximum at the point where the specimen is gripped in the jig (See Fig. 2(*a*)). A program was designed to study the shear strength of AZ80A-F alloy as a function of variations in: (*1*) root diameter, d; (*2*) $\left(\frac{s}{R}\right)$ ratio of support distance, s, between shear plane and the point of support to the radius, R, of the outside diameter of the specimen; (*3*) notch angle, α; and (*4*) notch radius, r.

Figure 3 shows that reducing the specimen diameter increases the shear stress. The same effect is reported above for double shear of both plain and notched specimens. Plotting both single and double shear results *versus* $\frac{s}{R}$ values (Fig. 8) for the various diameter specimens yields a family of curves when notch angle and notch radius are held constant. Good agreement is obtained with double shear data obtained on notched specimens. This implies that the single shear test with notched specimens is essentially the same as a double shear test with undercut and/or notched specimens. Figure 8 is a plot of the results in Table III (groups V, VI, VII, and VIII), and Table IV, showing the effect of varying $\frac{s}{R}$ on the shear strength for different specimen diameters. The curve shape was determined using the single shear data and the correlation analysis of Ezekiel,[7] while the position of the curves was determined with the aid of cross plots.

Increasing the $\frac{s}{R}$ ratio increases the shear stress to an optimum at $\frac{s}{R} = 0.3$. At higher $\frac{s}{R}$ values a gradual decrease in shear strength is observed. These results are not in agreement with the implica-

TABLE V.—SINGLE SHEAR TEST RESULTS FOR LOT II AZ80A-F EXTRUSIONS WITH VARYING NOTCH ANGLE AND NOTCH RADIUS.

Notch Angle, deg	r, in.	Shear Stress, 1000 psi. $s = 0.193$ $\frac{s}{R} = 0.616$			
		Minimum Diameter = 0.250 in.			Avg.
30	0.010	25.0	25.2	25.5	25.2
30	0.030	28.4	28.0	29.5	28.6
45	0.030	28.6	28.4	28.0	28.3
60	0.010	25.3	24.3	25.7	25.1
60	0.030	27.5	27.7	26.8	27.3
60	0.070	28.5	28.8	28.7	28.7
90	0.030	28.6	28.8	29.1	28.8

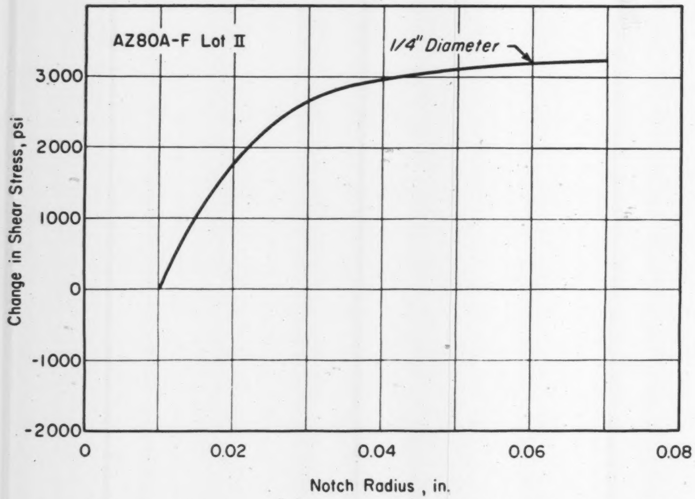

FIG. 9.—Change in Shear Stress Resulting from Change in Radius of Single Shear Specimens for $\frac{1}{4}$ in. Diameter.

tions of Gatto's findings. Gatto's photoelastic analyses indicate that the $\frac{s}{R}$ ratio must be maintained at one or greater to guarantee the absence of stress concentrations in the shear plane and implies that the optimum shear stress will be obtained under these conditions. Since the optimum shear stress in this investigation is found at an $\frac{s}{R}$ value of 0.3 a more complex stress system may be operating in the plastic range than was anticipated by Gatto. Others are invited to study this problem to determine if the true ultimate shear stress is obtained at an optimum $\frac{s}{R}$ value. The above correlation analysis, included the data from Table V, and indicates that variations in notch angle from 30 to 90 deg have no apparent effect on shear strength but that variations in notch radius can have a significant effect on the shear stress. Changing the notch radius from 0.010 in. to 0.040 in. increases the shear stress approximately 3000 psi (Fig. 9). The above statement on the effect of notch angle is in agreement with the findings of Gatto. However, he does state that notch radius does not influence the stress distribution in the shear plane from a practical standpoint, again implying that notch radius does not influence shear strength. Possibly Gatto's tests were conducted on specimens with notch radii of 0.040 in. or larger which minimize the effect and make it difficult to detect.

SINGLE SHEET SHEAR AND PUNCH SHEAR

Apparatus and Procedure:

The sheet specimen, when prepared as shown in Fig. 2(*b*), is mounted in the testing machine by clevis pins through each end. Shims were used in the clevises when necessary to prevent the possibility of rotation or lateral displacement of the specimen, and to insure axial loading.

TABLE VI.—SINGLE SHEAR AND PUNCH SHEAR STRENGTHS FOR AZ31A-O ALLOY.
Machined from ½ in. thick plate.

Nominal Thickness, in.	Single Shear Direction	W/2, in.	y, in.	Shear Stress, 1000 psi				
				Single Shear			Average	Punch Shear 2 in. Circumference
0.064......	transverse	0.750	0.170	26.7	26.6	25.5	26.3	19.7
0.125......	transverse	0.750	0.170	26.0	25.0	25.5	25.5	19.5
0.125......	longitudinal	0.750	0.170	25.7	25.8	23.8	25.1	...
0.187......	transverse	0.750	0.170	24.2	26.0	25.8	25.3	19.2
0.187......	longitudinal	0.750	0.170	25.6	25.5	25.8	25.6	...
0.250......	transverse	0.750	0.170	26.0	25.0	25.0	25.3	18.6
0.250......	longitudinal	0.750	0.170	24.2	24.1	24.5	24.3	...
0.375......	transverse	0.750	0.170	22.0	23.6	25.4	23.7	18.2
0.375......	longitudinal	0.750	0.170	23.6	24.0	21.3	23.0	...
0.500......	transverse	0.750	0.170	24.6	25.0	22.5	24.0	18.2
0.500......	longitudinal	0.750	0.170	24.4	24.7	24.4	24.5	...

Punch shear tests were performed with a punch and die having a circumference of 2 in.

TESTS AND RESULTS

Sheet structures analyzed for shear conditions, as a shear resistant web, are usually designed from punch shear data. However, punch shear values can be significantly different from single shear values as shown in Table VI. This difference may be attributable to stress concentration and/or anisotropy. This latter possibility would make it important to know the true shear strength in the same direction and plane as that shear which is induced in the structure. Since the direction of punch shear is always taken in the short transverse direction, these values would be of little value in materials having anisotropic shear properties if the design shear direction is in a longitudinal or transverse direction. These latter types of shear are actually encountered in all shear webs.

The method of Davis, Troxell, and Wiskocil[4] has been suggested by some investigators to obtain the shear strength of sheet in the desired direction. In essence the test is the same as for the single shear of round bars as previously described, with the specimen itself acting as the jig.

A program was designed to study the changes in shear strength as a function of directionality, sheet thickness, ratio of distance between holes to the half width of the specimen, $\frac{y}{w/2}$, and hole diameter. Punch shear tests were also made for comparison purposes. The range of useful values for $\frac{y}{w/2}$ is very limited. From a practical standpoint a very short distance between the holes makes it difficult to mount the specimen for testing without damaging it. From a theoretical standpoint the simple ratio of

$$\frac{\text{ultimate shear strength}}{\text{bending yield strength}}$$

calculated for the section must be greater than 1 to insure a shear failure. For wrought magnesium and aluminum a "*y*" value of 0.170 in. is satisfactory. For much smaller values of "*y*" the problem of fragility becomes noticeable. For a distance y of 0.170 in. a practical minimum width $\frac{W}{2}$ is about $\frac{3}{4}$ in.

The hole size in the range of $\frac{1}{16}$-in. to $\frac{1}{8}$-in. diameters has no apparent influence on shear strength. Distortion resulting from bending prior to shear was apparent in the specimens with larger size holes, therefore subsequent investigations were conducted with $\frac{1}{16}$-in. diameter holes.

The results of varying thickness are shown in Table VI. All magnesium specimens taken from $\frac{1}{2}$-in.-thick AZ31A-O plate were effectively center samples since equal amounts were machined from each side. Again a decrease in specimen size gives an increase in apparent shear stress (Fig. 10). The curve showing the effect of sheet thickness on the punch shear strength has the same curvature as that for the results of the single shear strength except that the punch shear curve is about 6000 psi lower. The sheet single shear strength results show somewhat larger scatter

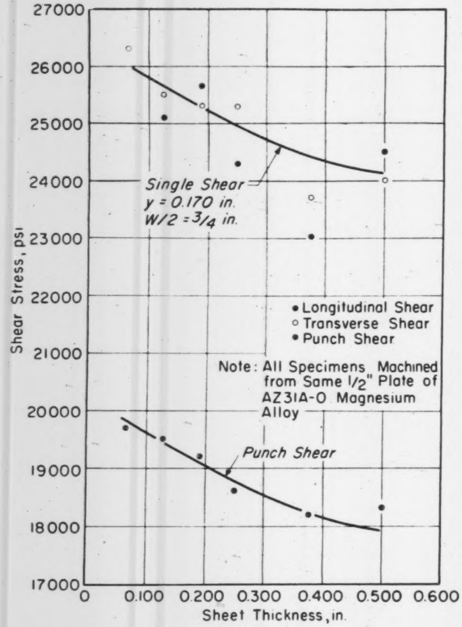

FIG. 10.—Sheet Single Shear Stress and Punch Shear Stress of AZ31A-O Magnesium Alloy as a Function of Sheet Thickness

TABLE VII.—SINGLE SHEAR AND PUNCH SHEAR STRENGTHS FOR ALUMINUM ALLOYS.

Alloy	Nominal Thickness, in.	Single Shear Direction	$W/2$, in.	y, in.	Shear Stress, 1000 psi	
					Single Shear	Punch Shear, 2 in. Circumference
2024-T3 clad.......	0.064	longitudinal	0.750	0.155	43.2	43.2
	0.064	longitudinal	0.750	0.155	42.9	43.0
	0.064	longitudinal	0.750	0.155	43.7	
	0.064	longitudinal	0.750	0.155	44.1	43.1 Avg.
	0.064	longitudinal	0.750	0.155	42.0	
	0.064	longitudinal	0.750	0.155	44.1	
	0.064	longitudinal	0.750	0.155	44.4	
	0.064	longitudinal	0.750	0.155	43.5 Avg.	
7075-T6 bare......	0.064	longitudinal	0.750	0.155	50.2	50.3
	0.064	longitudinal	0.750	0.155	48.9	50.2
	0.064	longitudinal	0.750	0.155	51.4	
	0.064	longitudinal	0.750	0.155	50.8	50.25 Avg.
	0.064	longitudinal	0.750	0.155	50.7	
	0.064	longitudinal	0.750	0.155	49.0	
					50.1 Avg.	

than that of single shear results for round specimens.

Longitudinal and transverse shear directions appear to have equal shear strength based on these data.

For two aluminum alloys, 2024-T3 clad and 7075-T6 bare, a series of specimens were tested, and the results are shown in Table VII. No difference between single shear strength and punch shear strength was noted for these alloys when compared on the basis of equal thickness rather than equal area, thereby suggesting that the short transverse shear strength is significantly higher than the longitudinal shear strength.

Punch shear results on the AZ80A-F, lot *I*, also show a significant size effect similar to that shown in Fig. 10 for the sheet. Average results of duplicate tests are 23,900 and 20,200 psi for $\frac{1}{8}$-in. and $\frac{1}{2}$-in. thick specimens, respectively.

CONCLUSIONS

All shear methods investigated show significant variations in apparent shear strength with changes in size of the shear specimens, the smaller specimens yielding the higher shear strengths. The conventional double shear method can be recommended for the testing of rivets or rivet wire since the application approximates the stress concentrations encountered in the test.

In designing for applications such as shear-resistant webs, or sheet fastener tear-out, the single shear results (round specimens or sheet) or notched double shear results are recommended over those obtained by punch shear, provided round specimens are carefully and properly oriented with respect to directionality when tested. The notched specimens with an $\frac{s}{R}$ value of 0.3 in either the single shear or double shear methods appear to give the best approach to the true shear strength. Comparisons of the shear strengths of material should only be made when the materials are tested under exactly the same conditions. Specimen size and geometry, jig geometry, tolerances and clearances, and wear of the shearing plates are all significant variables in shear testing but can be satisfactorily controlled with reasonable care.

Acknowledgment:

The authors wish to thank the Materials and Process Group of the Research Section in the Engineering Department of North American Aviation, Inc. for supplying the reference of Davis *et al.*,[4] and for their comments concerning their experience with the sheet single shear test method.

APPENDIX

EXPLANATION OF SYMBOLS

d = minimum pin diameter

T = tolerance between shear pin and holes in shearing plates (double shear jig)

t = center shear plate thickness (double shear jig)

S = shim thickness

s = distance from shear plane to pin support

R = $\frac{1}{2}$ maximum diameter of pin

N = notch depth

C = distance between outside shearing plates minus center shearing plate thickness (double shear jig)

r = notch radius

α = notch angle

W = width of sheet specimen

y = distance between holes in sheet specimen

DISCUSSION

MR. H. W. KLEINDIENST.[1]—In Fig. 3, the three *s/R* curves, 0.6, 1.0, and 0.9 do not appear to be in a sequence. The 1.0 seems to be out of place. Is there an explanation for that?

MR. R. W. FENN, JR. (*author*).—The value of 0.9 is a transcription error in the preprint. It should read 0.09. Reference to Fig. 8 shows the relationship of *s/R* ratio to shear values more clearly.

MR. KLEINDIENST.—Is it possible to describe the machining of the sheet specimen? Are there any special precautions to be taken, particularly at the holes?

MR. FENN.—The holes were drilled with sharp drills and the aid of a steel template for alignment. No further finishing was utilized.

MR. KLEINDIENST.—Figure 6 shows some shear stresses which begin roughly, they appear to be exactly at zero clearance. Were these tests made with any lubrication in the joints? Would a wet or dry lubricant make a difference in the results?

MR. FENN.—There was no lubricant used in these particular tests. However, this probably would be a desirable procedure.

MR. LAWRENCE S. LAZAR.[2]—Is there a notch sensitivity effect, as noticed in fatigue testing, in the notch shear test? In his paper the authors mentioned varying the depth and radius of the notch. In this respect, was any work done with samples with very large radii?

MR. R. B. CLAPPER (*author*).—The curve from Fig. 9 indicates a notch effect with change in radii from 0.01 in. to about 0.06 in. radius. However, it is not clear whether the change in shear stress was the result of notch effect, bending effect, or a combination of both. A valid proof test to determine this is difficult to design.

In the single-shear testing of the notched pins, radii were tested up to 3 in., but it was quickly determined that if radii exceeded approximately $\frac{1}{16}$ in., the failure could no longer be identified as a shear fracture. This was the result of bending stresses becoming significant a short distance from the shear-force centerline. Deflection of the bar resulted, thereby rotating the original shear plane, unless the pin was stiff enough to prevent anything but small deflections.

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[1] Design Specialist, Engineering Research Section, Republic Aviation Corp., Farmingdale, Long Island, N. Y.

[2] General Engineering Laboratory, General Electric Co., Schenectady, N. Y.

CREEP-RUPTURE TESTS IN SHEAR OF CAST ANTIMONY-LEAD ALLOYS

By J. Neill Greenwood[1]

Synopsis

A creep-rupture test has been developed in which a cylindrical pin of the alloy is sheared. The pins were machined from cast bars. Tests have been carried out over a temperature range from 0 to 100 C on alloys containing up to 11 per cent antimony. A specially refined industrial lead (AAG) was used as the basis of the alloys. Short-time tension tests (straining rate 0.6 per cent per min) have also been made.

The results have been plotted with stress against log time to fracture. Under short-time conditions the alloys appear much stronger than the pure lead. They rapidly lose this advantage as the life is increased by lowering the stress, the ratio for the 6 to 11 per cent alloy falling from 5:1 (short-time tension test) to 2:1 (stress for 30 yr life).

The work arose from the failure under field conditions of the sealing ring (11 per cent antimony-lead alloy) in an insulator on a power transmission line. The seal unites the porcelain insulator to a malleable iron cap and is cast into position. The annular seal is subjected to shear stresses imposed by the weight of the transmission cable. Some of these seals failed after about 12 months service. A tension test had been quoted as the basis for determining the load-carrying capacity.

As the power line crossed country subjected to high sun temperatures, particularly in the stagnant atmosphere of enclosed gullies, the experiments were carried out over a wide range of temperatures, namely 0 to 100 C.

Materials and Methods

The alloys were made from specially refined lead of the following composition:

Copper, per cent	0.0003
Bismuth, per cent	0.0001
Antimony, per cent	0.0001
Iron, per cent	0.0001
Zinc, per cent	0.0001
Sulfur, per cent	0.00005

The antimony used was Merck's ("extra pure"). It was dissolved in the lead at 400 C and poured into a steel mold kept at 180 C. The rods were $\frac{5}{16}$ in. in diameter and were turned to 0.25 in. (A few confirmatory tests were made with the 6 per cent alloy cast to $\frac{9}{16}$ in. diameter and turned to 0.5 in. No significant difference was found).

The alloys contained 1, 6, and 11 per cent antimony, respectively. Many casts of each nominal composition were made, and individual casts were checked by chemical analyses.

The shearing unit consisted of two self-aligning links, the male part having

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[1] Research Professor of Metallurgy, Baillieu Laboratory, University of Melbourne, Australia.

TABLE I.—VICKERS DIAMOND HARDNESS NUMBERS (DIAMOND PYRAMID HARDNESS).

Antimony, per cent	Range of Values	Average
Nil	3.5 to 4.2	4.0
1.0	6.3 to 7.8	7.0
6.0	12.0 to 15.5	13.5
11.0	15.5 to 18.0	16.5

TABLE II.—AGE HARDENING OF 6 PER CENT ANTIMONY ALLOY.

Time	Range of Values, D.P.H. Number	Average D.P.H. Number
As cast	11 to 12.5	11.5
1 day	10 to 12	10.5
2 days	10.5 to 12	11.5
3 days	11 to 12	12
7 days	12 to 13	12.5
14 days	12 to 13	12.5
28 days	14 to 16.5	15
60 days	15 to 17.5	16

a clearance of 0.001 in. The test pin connected the links, and loading was through a 12:1 simple lever.

For the tests at 0 C, the link assemblage was surrounded by melting ice. For temperatures higher than atmospheric the units were placed in a box heated by a series of 15-w bulbs. The energy supply was controlled by a simmerstat.

Tension test specimens were cast to shape in a steel mold. The castings were $\frac{1}{4}$ in. thick and were planed on both sides down to 0.15 in. thick. The specimens were provided with 1-in. gage lengths but as elongations were irregular they have not been reported here. The test specimens were strained at approximately 0.6 per cent per min, and the load could be read to the nearest 1 lb. The testing machine had been constructed especially for this kind of

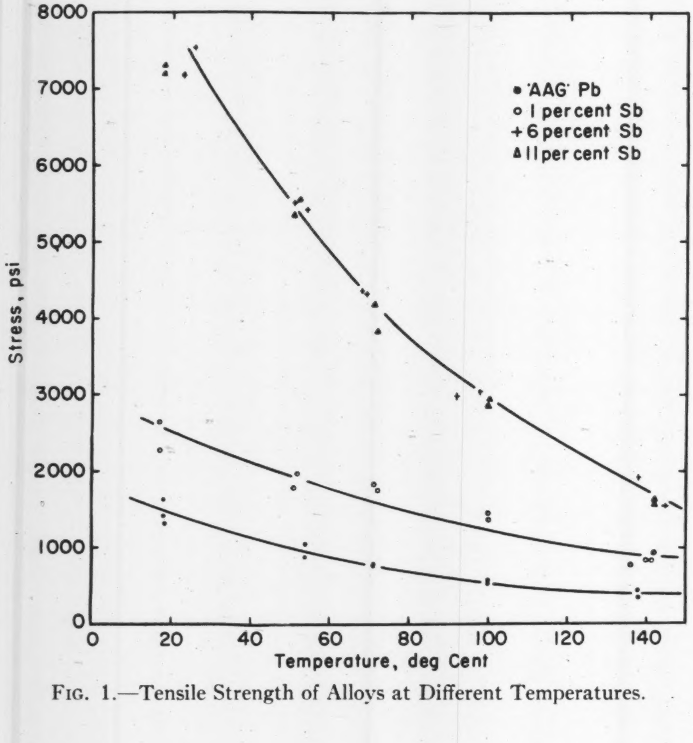

Fig. 1.—Tensile Strength of Alloys at Different Temperatures.

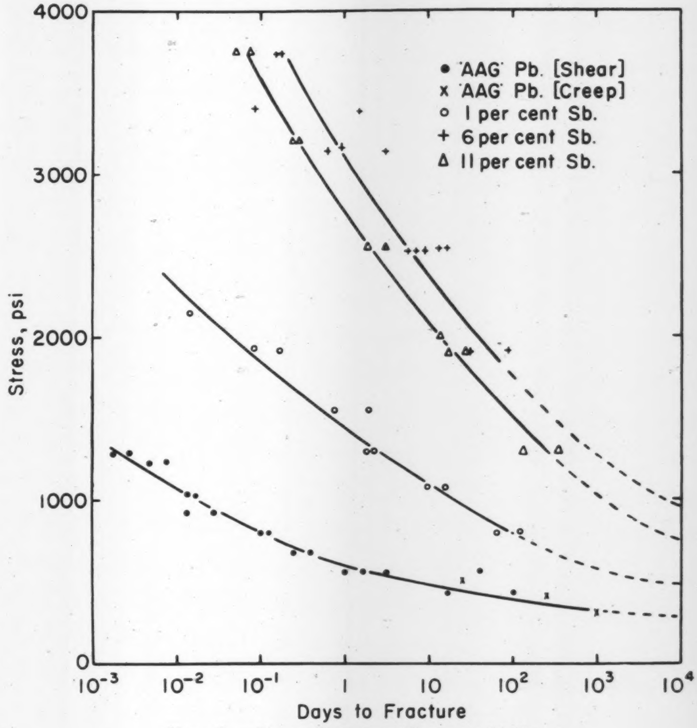

FIG. 2.—Creep-Rupture Tests at 22 C.

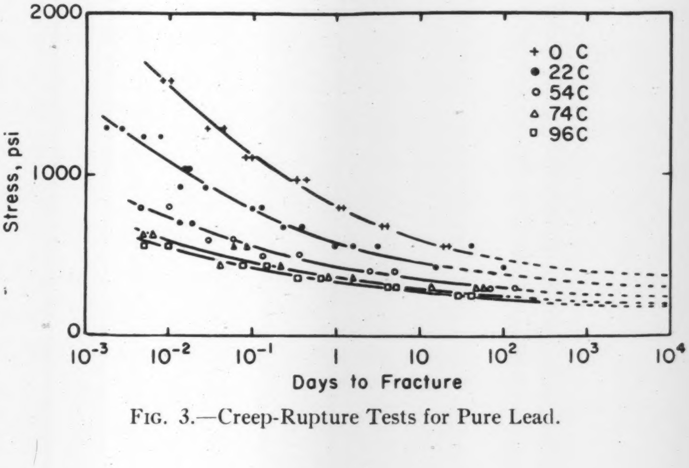

FIG. 3.—Creep-Rupture Tests for Pure Lead.

work, and the ambient temperature could be controlled by an enclosed heating element.

Hardness tests were made with the Vickers diamond indentor on cast machined tension test specimens. The load was 2.5 kg and the duration of loading 15 sec. The temperature was 20 ± 2 C.

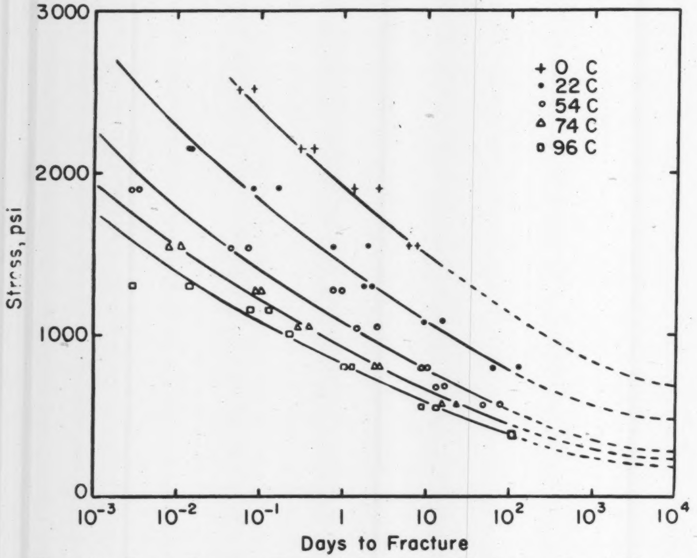

FIG. 4.—Creep-Rupture Tests for 1 per cent Antimony-Lead Alloy.

TEST RESULTS

Hardness:

The results given in Table I are from several specimens of each alloy. It will be seen that there is a general increase with antimony content. The solid solubility of antimony in lead falls from about 3.0 per cent at the eutectic temperature (247 C) to 0.24 per cent at 25 C. It is probable therefore that alloys containing more than 1.0 per cent of antimony will exhibit age hardening. Two separate castings of 6 per cent alloy were therefore tested for hardness over a period of 60 days. The results are given in Table II. It will be seen that no substantial hardening occurred until after 14 days.

Tensile Strength:

The values obtained for the several alloys are plotted in Fig. 1. The slope of the curves in the higher region of temperatures is such that the strength would be zero at the melting temperatures,—respectively 327, 300 and 247 C. It will be noted that at all temperatures the strength of the 6 per cent alloy is the same as that of the 11 per cent alloy. This is interesting in view of the hardness values given in Table I.

Creep-Rupture Tests:

The results of the creep-rupture tests are plotted in Figs. 2 to 6. In Fig. 2 a comparison is made of the three alloys with the basis lead at 22 C. On the curve for pure lead, three values marked by crosses were obtained in tensile-creep tests of the same lead extruded.

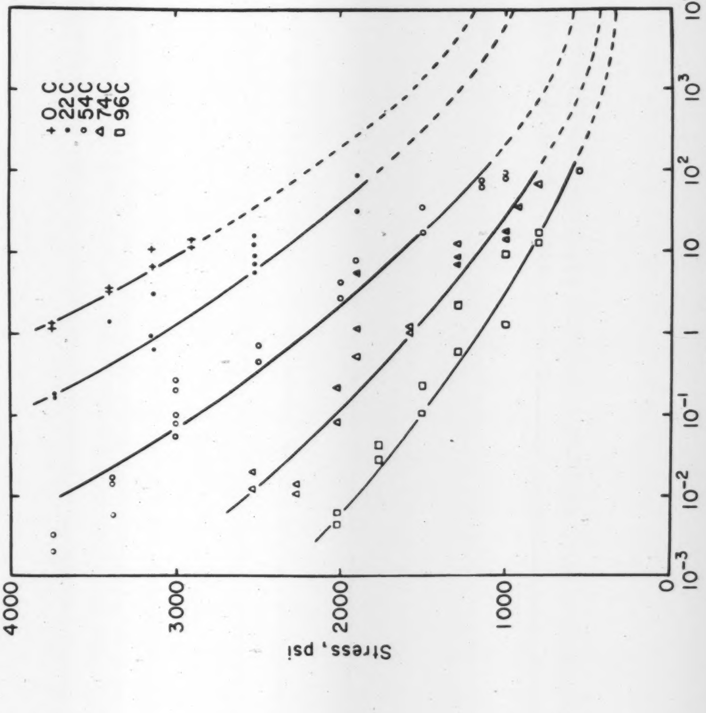

FIG. 5.—Creep-Rupture Tests for 6 per cent Antimony-Lead Alloy.

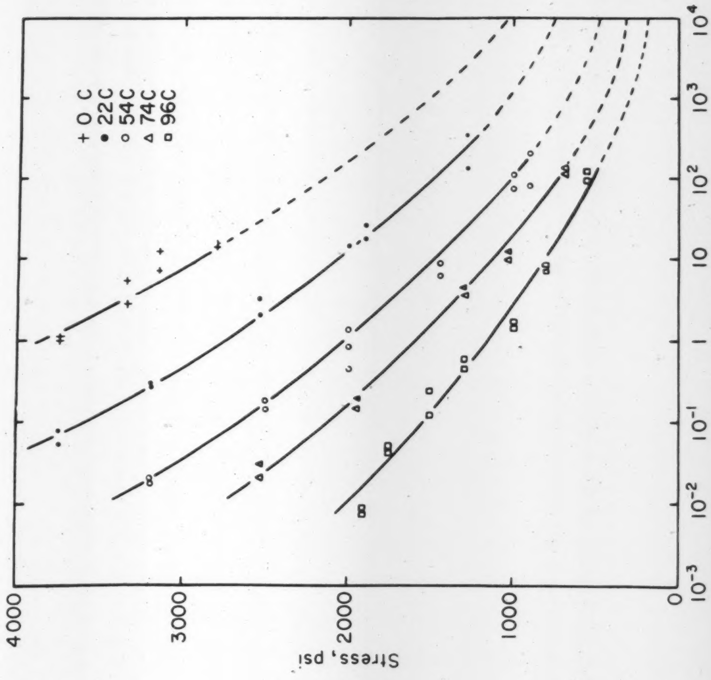

FIG. 6.—Creep-Rupture Tests for 11 per cent Antimony-Lead Alloy.

In drawing the curves for the alloys cognizance has been taken of the curvature of the relationship for pure lead. It would have been possible to draw straight lines through the points for the alloys, but this would lead to the unreasonable result of expecting failure in finite times under no stress.

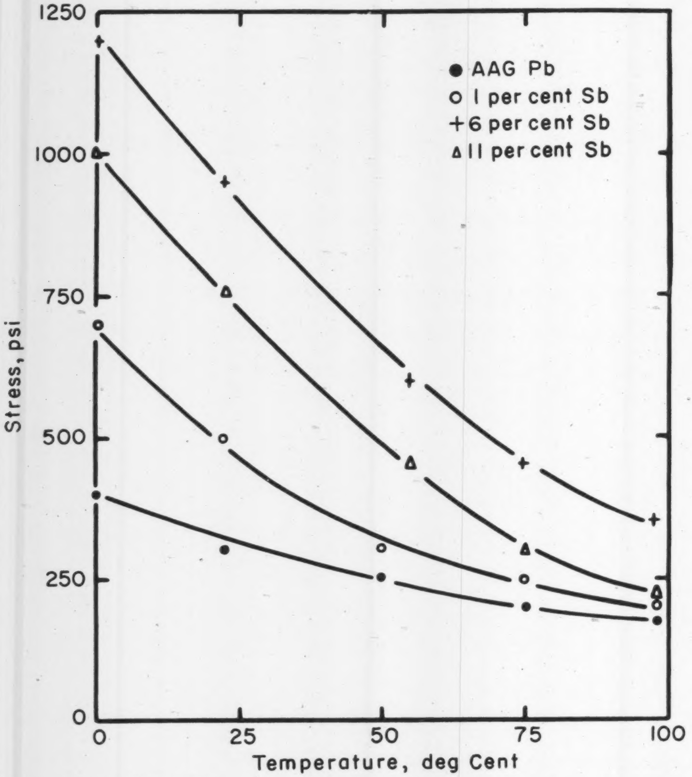

FIG. 7.—Stresses for Life of Ten Thousand Days (estimated) for Antimony-Lead Alloys at Different Temperatures.

With tests not extending beyond 100 days, the extrapolation of the curves is obviously somewhat arbitrary. The course of the curves has, however, been determined by following what seemed to be the natural curvature. The terminal stresses for 10^4 days so obtained were then plotted for the several temperatures in Fig. 7. It is assumed that, as in Fig. 1. these curves could be extrapolated to zero stress at the melting temperature for the respective alloys. In this way the terminal stresses for 10^4 days were smoothed out and the final extrapola-

TABLE III.—COMPARISON OF TENSILE STRENGTH AND STRESS FOR 30-YR LIFE.

Alloy	20 C (68 F)		50 C (122 F)	
	Tensile Strength, psi	Stress for 30 yr Life, psi	Tensile Strength, psi	Stress for 30 yr Life, psi
Pure lead........	1500	325	1000	250
1 per cent antimony.........	2600	500	2000	325
6 per cent antimony.........	7700	1000	5500	650
11 per cent antimony.........	7700	775	5500	500

tions made as shown in Figs. 3 to 6 in which the values for the respective alloys are given for a series of five temperatures.

SUMMARY

The results obtained have been used to estimate the maximum stress which could be permitted on the sealing rings of the insulator concerned. As in all such cases, the engineer must fix what he considers a reasonable life, having in mind the conditions to which the part will be subjected. As an example, values have been estimated for a life of about 30 yr. They would not be very different for 100 yr. These are given in Table III, and are compared with the tensile strength as determined in short-time tests.

From this comparison it will be seen how unreliable the tension test is in a case like this. The alloys containing 6 to 11 per cent antimony have a tensile strength approximately five times that of pure lead, yet the stress for a long life is only about double. Also where the alloys are to be used in ambient temperatures higher than atmospheric the stress which can be used in design falls by about one-third for a rise of 30 C.

It must be realized also, in applying the values of Table III, that the usual engineering practice of reducing the stresses by a factor of safety should be followed.

THE CORROSION PROPERTIES OF CHROMIUM-NICKEL-MANGANESE AUSTENITIC STAINLESS STEELS*

BY W. G. RENSHAW[1] AND R. A. LULA[1]

SYNOPSIS

The chromium-nickel-manganese steels have been developed originally as a conservation measure for nickel. Their mechanical and corrosion properties are very similar to the chromium-nickel grades and are therefore important in the present economic circumstances with dwindling nickel availability. This paper deals with the corrosion properties of the chromium-nickel-manganese steels, AISI 201 and 202, and also some lower carbon and higher chromium grades. The influence of individual elements chromium, nickel, manganese has been investigated first and then the corrosion properties of the commercial analysis were evaluated in various corrosion media. The corrosion properties of sensitized material has been studied and in relation with this the carbide and nitride precipitation.

Results of the investigation indicate that chromium content has a significant influence on general corrosion resistance, as shown by improvement of nitric acid resistance with increasing chromium to 20 per cent. Nickel provides an additional improvement in resistance to general corrosion, and is especially effective in promoting resistance to localized attack. Manganese exerts relatively little influence through the range 3 per cent to 14 per cent covered in this work.

Resistance of these steels to intergranular corrosion, like that of chromium-nickel steels, in the sensitized condition, can be increased by decreasing carbon content. Carbon is somewhat less critical in chromium-nickel-manganese steels and resistance to intergranular corrosion after short time sensitization is better than that of a chromium-nickel steel of the same carbon content.

There have been many attempts in the past to replace the nickel in austenitic stainless steels with manganese or with manganese and nitrogen, especially during nickel shortages. The chromium-nickel-manganese steels in which only half of the nickel has been replaced by manganese and nitrogen have been very successful in this respect; they combine good mechanical properties with corrosion resistance equivalent to the chromium-nickel grades while accomplishing a net saving in nickel. The industrial importance of these steels has been recognized and they are expected to find an ever expanding field of application since the rate of increase in nickel production is exceeded by the rate of increase in the production of nickel-bearing alloys.

Two of the chromium-nickel-manganese steels have been given the AISI designation type 201 and 202 following the procedure for the 300 series and there are three other grades which do not have, up to this time, an AISI designation but which we will call throughout this paper type 204, 204L and 20-6-8 based on the nomenclature used for the 300 series. The nominal composition of these steels is shown in Table I.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] The Authors are associated with the Allegheny Ludlum Steel Corp. Research Lab. Brackenridge, Pa.

TABLE I.—COMPOSITION OF CHROMIUM-NICKEL-MANGANESE STEELS, PER CENT.

Designation	Carbon	Manganese	Chromium	Nickel	Nitrogen
AISI 201..........	0.15 max	5.50 to 7.50	16.0 to 18.0	3.5 to 5.5	0.25 max
AISI 202..........	0.15 max	7.50 to 10.0	17.0 t o19.0	4.0 to 6.0	0.25 max
204[a].............	0.10 max	7.50 to 10.0	17.0 to 19.0	4.0 to 6.0	0.25 max
204L[a]............	0.06 max	7.50 to 10.0	17.0 to 19.0	5.0 to 7.0	0.25 max
20-6-8............	0.10 max	7.0 to 9.0	19.0 to 21.0	5.0 to 7.0	0.25 to 0.35

[a] Not standard AISI type.

In spite of the fact that these steels are not new, only a limited amount of data on their corrosion properties is available. Franks, Binder, and Thompson[2] have reported boiling 65 per cent nitric acid data and 10 per cent sulfuric acid results for experimental chromium-nickel-manganese heats with 15 to 18 per cent chromium.

It is the purpose of this paper to evaluate the corrosion resistance of chromium-nickel-manganese steels under strongly oxidizing conditions, under intermediate corrosive environments, and under localized conditions, and to emphasize the influence which chromium, nickel, and manganese exert. Thus the data presented here should provide valuable knowledge regarding the standard types 201 and 202 as well as indicate some composition with improved corrosion resistance for certain more severe environments.

Since resistance to intergranular corrosion is vitally important, especially in applications involving either welding or stress relieving, this investigation was extended to sensitized material in order to find the influence of carbon and nitrogen in wholly austenitic chromium-nickel-manganese steels.

MATERIALS

The steels used in this investigation were either from large production heats or from laboratory experimental heats. The production heats, designated by five-digit numbers, are all in the composition range shown in Table I. The laboratory heats are of standard composition and also of nonstandard composition such as required to determine the influence of various elements on the corrosion properties.

In order to be able to hot-roll large ingots of austenitic steels they have to be austenitic up to approximately 2300 F. For this reason, the composition of the steels used throughout this paper was balanced in order to have a completely austenitic structure at least up to 2300 F. A high nitrogen content is required in all chromium-nickel-manganese steels for this purpose. Whenever a small amount of delta ferrite was present, in the case of an experimental heat with low nickel, it was so specified.

All the specimens were taken from sheet 0.06 to 0.10 in. thick, annealed at 1950 F followed by air cooling.

METHODS

To obtain complete knowledge of the behavior of the stainless steels under a wide variety of corrosive environments by laboratory tests would be an endless task. However, certain selected tests have proven valuable in the investigation of new materials. Among these the boiling 65 per cent nitric-acid test represents strongly oxidizing acid conditions. Its use can also illustrate the contribution of individual elements like chromium, nickel, and manganese on the corrosion resistance. All materials covered in this discussion have been tested in this medium. Specimens 1 by 2 in. were prepared from annealed sheet material with all of the edges machined to remove shear strain. The procedure followed was that covered in ASTM Recommended Practice A 262.[3] In addition, sensitized samples were tested by this method in a study of resistance to intergranular corrosion, and also in the copper sulfate - sulfuric acid solution in accordance with ASTM Recommended Practice A 393.[4]

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[2] R. Franks, W. O. Binder and J. Thompson, "Austenitic Chromium-Manganese-Nickel Steels Containing Nitrogen," *Transactions*, Am. Soc. for Metals, Vol. 47, (1954).

For intermediate conditions, a boiling glacial acetic acid test and a boiling 10 per cent phosphoric acid solution have been used to illustrate neutral or mildly oxidizing acid environments. In both of these, type 304 shows very good resistance, and any material which exhibits high rates of attack under these conditions would probably be resistant only in very mild environments. The boiling glacial acetic test was conducted for five 48-hr periods in flasks fitted with reflux condensers. In some instances specimens were placed in the vapors as well as in the liquid to determine susceptibility to pitting in acetic vapor. The boiling 10 per cent phosphoric acid test involved 2-hr exposure to 10 per cent by weight solution in reflux condensers. All specimens were activated in warm (140 F) hydrochloric acid solution prior to the tests, so that the chromium-nickel-manganese steels could exhibit their ability to become passive under mildly oxidizing or neutral conditions.

This inherent ability to attain a passive condition is governed by composition and conditions of exposure. When artificially activated specimens are used in tests, it usually can be assumed that the metal should be completely resistant to attack if they become passive and remain so during the test in the corrosive medium.

Other intermediate category solutions used for tests on selected heats included concentrated sulfurous acid liquid and vapors at room temperature, and 10 per cent citric acid at 200 F. Aggressive organic acids of a reducing and neutral nature were represented by 10 per cent oxalic acid at 200 F and boiling 50 per cent lactic acid, respectively. Specimens 1 by 2 in. were employed in all tests. Specimens were exposed for 2 hr in the hot solutions whereas the sulfurous acid exposure was continued for 28 days. All of these tests represent specialized cases where organic acids of oxidizing or neutral nature, or inorganic acid of a reducing nature are involved. In some of these, such as sulfurous acid vapor, 18-8 steels may be subject to pitting, and the test was selected to learn whether steels with high manganese might offer better corrosion resistance. No aeration or agitation was used in any of these.

For the study of the resistance to localized attack, the 20 per cent salt spray and boiling 42 per cent magnesium-chloride tests were employed. While neither of these tests produce results which can be compared numerically, the salt-spray test can be useful in illustrating the influence of various elements on the degree of resistance to pitting by chlorides.

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[3] Tentative Recommended Practice for Boiling Nitric Acid Test for Corrosion-Resisting Steels (A 262 - 55 T), 1955 Book of ASTM Standards, Part I, p. 1108.

[4] Tentative Recommended Practice for Conducting Acidified Copper Sulfate Test for Intergranular Attack in Austenitic Stainless Steel (A 393 - 55 T), 1955 Book of ASTM Standards, Part I, p. 1135.

TABLE II.—INFLUENCE OF NICKEL ON NITRIC ACID CORROSION RATES.

Heat	Composition, per cent					Corrosion Rate, in. per month					
	Carbon	Chromium	Manganese	Nickel	Nitrogen	1st	2nd	3rd	4th	5th	Average of five
ED40	0.11	17.13	10.17	1.97	0.20	0.0038 0.0036	0.0034 0.0035	0.0035 0.0035	0.0032 0.0031	0.0029 0.0029	0.0034 0.0033
EX882	0.14	17.17	9.05	2.94	0.12	0.0045 0.0041	0.0043 0.0042	0.0043 0.0051	0.0048 0.0047	0.0049 0.0046	0.0046 0.0046
ED41	0.091	17.22	9.99	3.05	0.22	0.0028 0.0029	0.0027 0.0027	0.0027 0.0027	0.0026 0.0026	0.0027 0.0027	0.0027 0.0027
DG23	0.12	18.24	10.08	3.20	0.16	0.0026 0.0026	0.0021 0.0021	0.0019 0.0019	0.0018 0.0018	0.0019 0.0019	0.0019 0.0019
DF77	0.056	17.83	9.89	4.06	0.13	0.0021 0.0018	0.0019 0.0021	0.0019 0.0018	0.0016 0.0016	0.0016 0.0016	0.0018 0.0018
18007	0.089	17.75	9.14	5.28	0.14	0.0016 0.0016	0.0017 0.0017	0.0017 0.0017	0.0015 0.0016	0.0016 0.0016	0.0016 0.0016
25350	0.024	17.22	9.99	5.39	0.20	0.0016 0.0017	0.0016 0.0016	0.0016 0.0016	0.0015 0.0016	0.0017 0.0016	0.0016 0.0016
DG65	0.10	17.74	6.45	6.28	0.14	0.0016 0.0017	0.0014 0.0015	0.0013 0.0013	0.0011 0.0013	0.0011 0.0010	0.0013 0.0014
DG63	0.044	17.72	6.66	6.28	0.15	0.0014 0.0014	0.0013 0.0013	0.0011 0.0011	0.0019 0.0011	0.00087 0.0011	0.0013 0.0012
ED42	0.10	19.60	10.17	1.99	0.31	0.0022 0.0023	0.0021 0.0022	0.0020 0.0021	0.0019 0.0020	0.0020 0.0020	0.0020 0.0021
ED45[a]	0.090	20.52	10.28	2.04	0.31	0.0019 0.0019	0.0018 0.0018	0.0019 0.0019	0.0019 0.0019	0.0018 0.0018	0.0019 0.0019
DT99	0.10	20.05	10.26	4.18	0.39	0.0014 0.0013	0.0012 0.0012	0.0011 0.0012	0.0012 0.0012	0.0011 0.0011	0.0012 0.0012
DX24	0.041	20.10	8.87	3.94	0.36	0.0012 0.0012	0.0011 0.0011	0.0011 0.0012	0.0012 0.0012	0.0014 0.0014	0.0012 0.0012
DU17	0.095	19.97	9.32	5.64	0.40	0.0013 0.0012	0.0011 0.0012	0.00098 0.0010	0.0011 0.0011	0.0010 0.0010	0.0011 0.0011
DU21	0.030	20.01	9.00	5.50	0.32	0.0011 0.0010	0.00080 0.00085	0.00084 0.00083	0.00081 0.00080	0.00082 0.00083	0.00087 0.00087

[a] Approximately 3 per cent delta ferrite in the annealed condition.

The boiling magnesium-chloride test is capable of determining whether any given composition is resistant to stress corrosion cracking.

The 20 per cent salt-spray test involved exposure to a 20 per cent by weight salt fog at 95 F. All samples were placed at 15 deg. from the vertical, facing the source of the spray for 100 hr. Specimens 6 by 6 in. were used.

For the study of stress corrosion, boiling 42 per cent by weight magnesium chloride (boiling point 309 F) was employed, using reflux condenser assemblies. Specimens 1 by 6 in. were bent into a U-shape and stressed by applying tension with bolts through holes located $\frac{1}{2}$ in. from each end.

Not all compositions were tested under all conditions discussed above, but sufficient data have been obtained to show significantly the influence of various elements, and to point out suitable analyses for best corrosion resistance in different classes of environments. All solutions were prepared with cp salts or acids, and behavior may vary where contaminants are present.

INFLUENCE OF VARIOUS ELEMENTS

In order to investigate properties imparted by chromium, nickel, and manganese, each element was varied over certain ranges, keeping the other two at constant levels. This was done to justify the selection of specific analyses possessing optimum mechanical and corrosion-resistant properties.

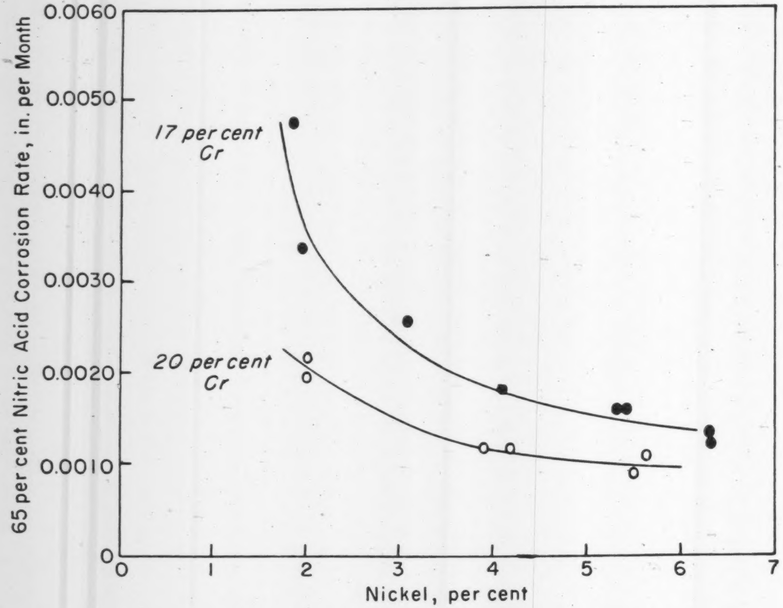

FIG. 1.—Influence of Nickel Content on Nitric Acid Corrosion Rates at 17 per cent and 20 per cent Chromium Levels.

Influence of Nickel:

Of the three elements which have been varied in this investigation, nickel appears to be very important for it has a beneficial effect in strongly oxidizing environments as well as under localized pitting conditions. The boiling nitric-acid test, which represents very severe oxidizing conditions, shows a marked im-

TABLE III.—INFLUENCE OF NICKEL ON CORROSION RATES UNDER INTERMEDIATE CONDITIONS AND IN ORGANIC MEDIA.

Heat	Composition, per cent					Boiling, Glacial-Acetic Acid in. per month		Sulfurous Acid, loss in 28 days		Boiling, 10 per cent Phosphoric Acid, in. per month	10 per cent Citric Acid, 200 F, in. per month	Boiling 50 per cent Lactic Acid, in. per month	10 per cent Oxalic Acid, 200 F, in. per month
	Carbon	Manganese	Chromium	Nickel	Nitrogen	Liquid	Vapor	Liquid	Vapor				
ED40	0.11	10.17	17.13	1.97	0.20	0.00005	0.00095	...	...	0.00045 0.00034	0.00056 ...	0.0460 ...	... ...
DG23	0.12	10.08	18.24	3.20	0.16	0.00011	0.00086	0.0002	0.0004[a]	Nil	...	...	...
DF78	0.054	9.45	17.37	4.04	0.057	0.00020	0.0012	0.0003	0.0006[a]	0.00043 ...	0.00025 0.00029	0.0369 ...	0.0250 0.0246
DG26	0.12	9.90	18.29	4.53	0.06	0.00025	0.0010	0.0004	0.0005	0.00022 ...	... ...	0.0329 ...	0.0243 0.0227
DG63	0.044	6.66	17.72	6.28	0.15	0.00006	0.0016[a]	0.0003	0.0003[a]	Nil ...	0.00025 0.00019	0.0295 ...	0.0234 0.0220
ED45	0.090	10.28	20.52	2.04	0.31	0.00010	...	...	...	Nil	0.00053	Nil	...
DX24	0.041	8.87	20.10	3.94	0.36	0.00003	...	0.0012	0.0002	Nil ...	... ...	... ...	0.0197 0.0225
DU21	0.030	9.00	20.01	5.50	0.32	0.00002	...	0.0005	0.0007	Nil ...	0.00017 0.00022	0.00013 0.00016	0.0271 0.0284
ED55	0.10	8.66	15.02	5.94	0.21	...	...	...	...	0.0863 0.0849	0.0060 ...	0.0792 ...	0.0206 0.0258
ED36	0.11	13.70	14.93	6.03	0.20	0.0030	...	...	...	0.136 0.134	0.0080 ...	0.1120 ...	0.0254 0.0267
98963	0.044	1.64	18.24	9.56		0.00011	0.0004	0.0004	0.0007	Nil	Nil	0.0804 0.0976	0.0065 ...

[a] Pitting.

provement in corrosion rates as nickel is increased from 2 per cent to 6 per cent. The results are given in Table II and plotted in Fig. 1, with chromium-nickel-manganese steels having 17 per cent and 21 per cent chromium.

Previous studies of chromium-manganese steels with 1 per cent nickel[5] have shown corrosion rates about 4 to 6 times those of 18-8 steels. It can be seen by inspection of Fig. 1 that at 17 per cent chromium, significant improvement can be obtained as nickel is increased from 2 per cent to 3 per cent. From this point the corrosion rates drop rapidly as nickel increases to 4 per cent and then decrease more slowly beyond this point. If this curve were extrapolated to 8 per cent, it would correspond closely to the value for type 302.

At the 20 per cent chromium level, the benefit of increasing nickel is not as pronounced, but in this case all corrosion rates are much lower due to the higher chromium. Data for the heat DU21 with 20 per cent chromium, 9 per cent manganese and 5.5 per cent nickel show it to be comparable to 18-8 steel in boiling nitric acid. This illustrates that a composition is available with corrosion resistance considerably better than that of type 202, and practically as good as that of type 304 under severe conditions usually extant in chemical industry applications. Such a material would at the same time save several per cent of nickel.

Where corrosive applications of a less severe nature are involved, data indicate that types 201 or 202 would be more than adequate under most circumstances. Heat DF77, a type 204 heat, has about twice the nitric acid corrosion rate of type 304, but it could still be expected that such a composition would show very good resistance to less aggressive media.

Table III summarizes data in intermediate conditions and in organic acids. The intermediate and nonoxidizing media typified by acetic, sulfurous, phosphoric, and citric acids produce no significant attack with the type 202 composition.

The influence of nickel on corrosion resistance in these intermediate nonoxidizing solutions is negligible in the composition limits covered. Even a 17 per cent chromium, 2 per cent nickel analysis (ED40) shows extremely low corrosion rates. In some of the stronger organic acids where the chromium-nickel-manganese steels are appreciably attacked, nickel variation still shows little influence on corrosion resistance. For example, within the limits of 2 per cent to 6 per cent nickel no improvement in corrosion resistance was obtained in hot oxalic solution. Even type 304 is subject to attack under these conditions. Chromium, nickel, manganese steels might be useful where type 304 has been successfully used with certain oxalic acid environments. In boiling lactic acid, there does not appear to be any advantage in increasing nickel from 2 per cent to 6 per cent when chromium content is 17 per cent. However, indications are that a high nickel and chromium content (heat DU21) improves resistance to this boiling lactic solution so that a composition even better than type 304 is available. Nickel alone cannot accomplish this as is evident in the case of 15 per cent chromium, 6 per cent nickel, manganese steels which are severely attacked.

The influence of nickel is important in cases where localized attack or pitting occurs. There are many such instances where type 304 can be employed successfully. The 20 per cent salt-spray test offers a comparison of behavior among chromium-nickel-manganese steels, and also with type 304 under conditions which are conducive to localized attack.

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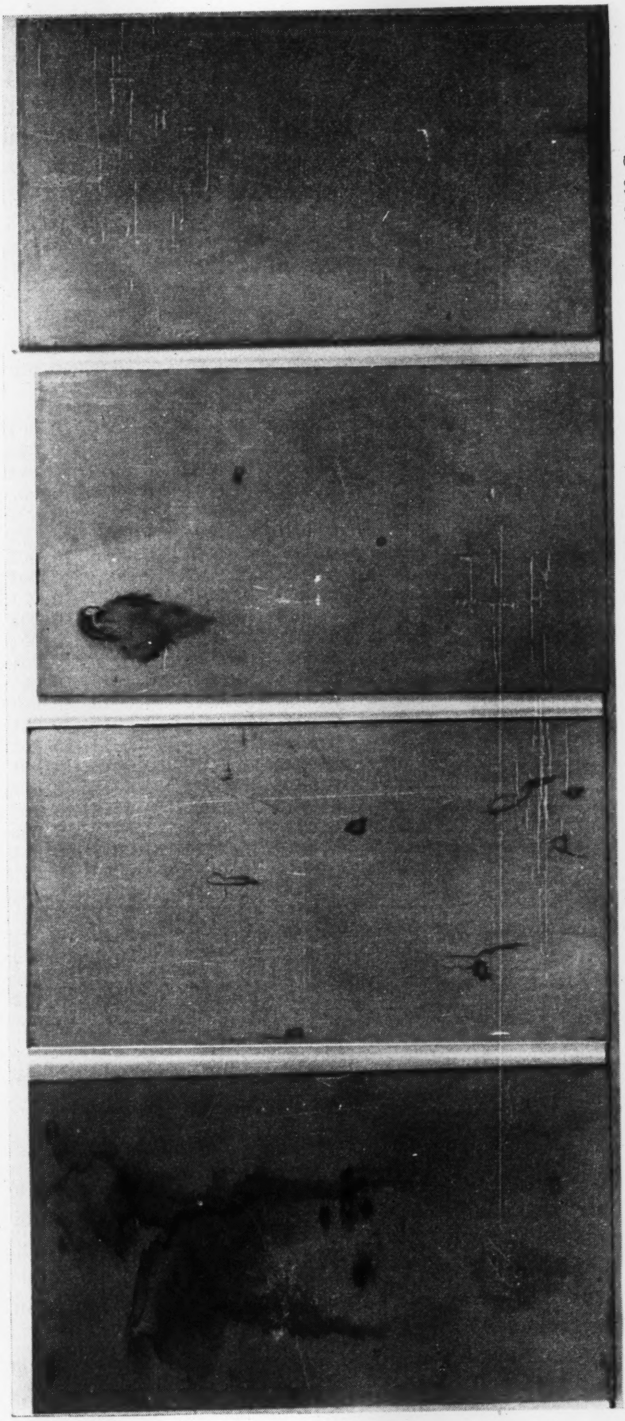
[5] R. A. Lincoln, "Nickel Restrictions Bring Use of New Stainless Steels," *Iron Age*, Vol. 171, No. 20, May 14, 1953, p. 129.



17.13 Cr
1.97 Ni
10.17 Mn

17.36 Cr
4.03 Ni
10.04 Mn

19.60 Cr
1.99 Ni
10.17 Mn

19.60 Cr
5.49 Ni
9.07 Mn

FIG. 2.—Influence of Nickel on Salt-Spray Resistance.

A photograph is best for summarizing the results. Figure 2 illustrates the influence of nickel in 17 per cent, chromium, 10 per cent manganese, nickel and 20 per cent chromium, 10 per cent manganese, nickel steels. The beneficial effect is much less evident at a 17 per cent chromium level than at 20 per cent chromium. Increasing nickel from 2 per cent (heat ED42) to 5.5 per cent (heat DY35) causes a pronounced improvement in salt-spray resistance, with behavior equalling type 304 under these same conditions of 100-hr exposure. It is emphasized, however, that nickel itself is not wholly responsible for salt-spray behavior. A 15 chromium, 6 nickel, 9 manganese heat (ED36) shown later in Fig. 6 is seriously pitted and attacked in the salt-spray test, pointing to the necessity for chromium also.

TABLE IV.—INFLUENCE OF MANGANESE ON NITRIC ACID CORROSION RATES OF 17 PER CENT CHROMIUM-NICKEL-MANGANESE STEELS.

Heat	Composition, per cent					Corrosion Rate, in. per month					
	Carbon	Manganese	Chromium	Nickel	Nitrogen	1st	2nd	3rd	4th	5th	Average of five
DM32	0.090	3.80	17.74	4.07	0.12	0.0019 0.0018	0.0018 0.0018	0.0015 0.0015	0.0016 0.0016	0.0015 0.0015	0.0016 0.0016
DM29	0.090	4.63	17.83	3.99	0.13	0.0017 0.0017	0.0015 0.0015	0.0017 0.0018	0.0017 0.0017	0.0015 0.0015	0.0016 0.0016
DM35	0.023	5.58	17.88	4.15	0.15	0.0017 0.0017	0.0016 0.0016	0.0014 0.0014	0.0015 0.0014	0.0014 0.0014	0.0015 0.0015
DF76	0.066	7.93	17.42	4.00	0.11	0.0018 0.0018	0.0017 0.0017	0.0016 0.0016	0.0015 0.0015	0.0015 0.0015	0.0016 0.0016
DF78	0.054	9.45	17.37	4.04	.057	0.0023 0.0023	0.0021 0.0021	0.0017 0.0017	0.0017 0.0017	0.0017 0.0017	0.0019 0.0019
DF77	0.056	9.89	17.83	4.06	0.13	0.0021 0.0018	0.0019 0.0021	0.0019 0.0018	0.0016 0.0016	0.0016 0.0016	0.0018 0.0018
59518	0.12	10.35	17.69	4.08	0.15	0.0022 0.0023	0.0018 0.0019	0.0017 0.0017	0.1018 0.0018	0.0019 0.0019	0.0019 0.0019
25311	0.10	10.04	17.36	4.03	0.13	0.0019 0.0020	0.0017 0.0018	9.0016 0.0017	0.0017 0.0017	0.0017 0.0018	0.0017 0.0018
DE20	0.092	11.00	17.41	3.66	0.18	0.0021 0.0021	0.0019 0.0020	0.0019 0.0019	0.0018 0.0018	0.0018 0.0018	0.0019 0.0019
DQ31	0.12	12.10	17.74	3.89	0.23	0.0024 0.0023	0.0020 0.0020	0.0020 0.0020	0.0019 0.0019	0.0021 0.0021	0.0021 0.0021
DQ47	0.10	13.44	17.28	3.97	0.16	0.0022 0.0023	0.0018 0.0019	0.0017 0.0017	0.0018 0.0019	0.0018 0.0018	0.0018 0.0019
DQ50	0.11	14.00	17.56	4.10	0.18	0.0023 0.0022	0.0020 0.0019	0.0018 0.0018	0.0018 0.0017	0.0020 0.0019	0.0020 0.0019
ED47	0.10	16.30	17.14	3.98	0.16	0.0029 0.0029	0.0025 0.0025	0.0025 0.0025	0.0025 0.0024	0.0023 0.0023	0.0025 0.0025
ED56	0.10	17.71	17.25	3.91	0.17	0.0030 0.0030	0.0026 0.0026	0.0027 0.0025	0.0024 0.0024	0.0026 0.0026	0.0027 0.0026

In general, results point to the same conclusions regarding nickel that usually are mentioned with chromium-nickel steels, namely, that it tends to promote or stabilize the passive state, and offers benefit under localized conditions of corrosion.

Influence of Manganese:

The question of manganese in these steels is obviously an important one, since this is the element which has been increased many times over the amount in which it is present in chromium-nickel steels.

The nitric acid data are given in Table IV and Fig. 3. Fourteen heats containing

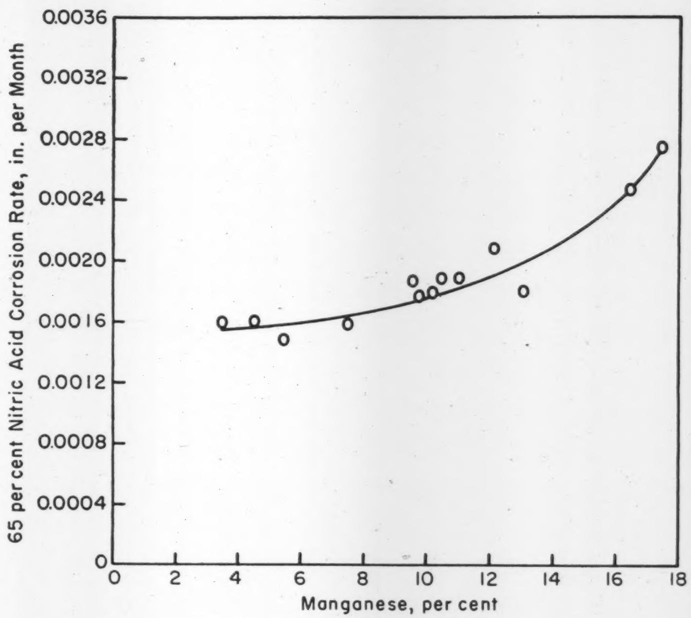


FIG. 3.—Influence of Manganese on Nitric-Acid Corrosion Rates of 17 per cent Chromium, 4 per cent Nickel, Manganese Steels.

TABLE V.—INFLUENCE OF MANGANESE ON CORROSION RATES UNDER INTERMEDIATE CONDITIONS AND IN ORGANIC MEDIA.

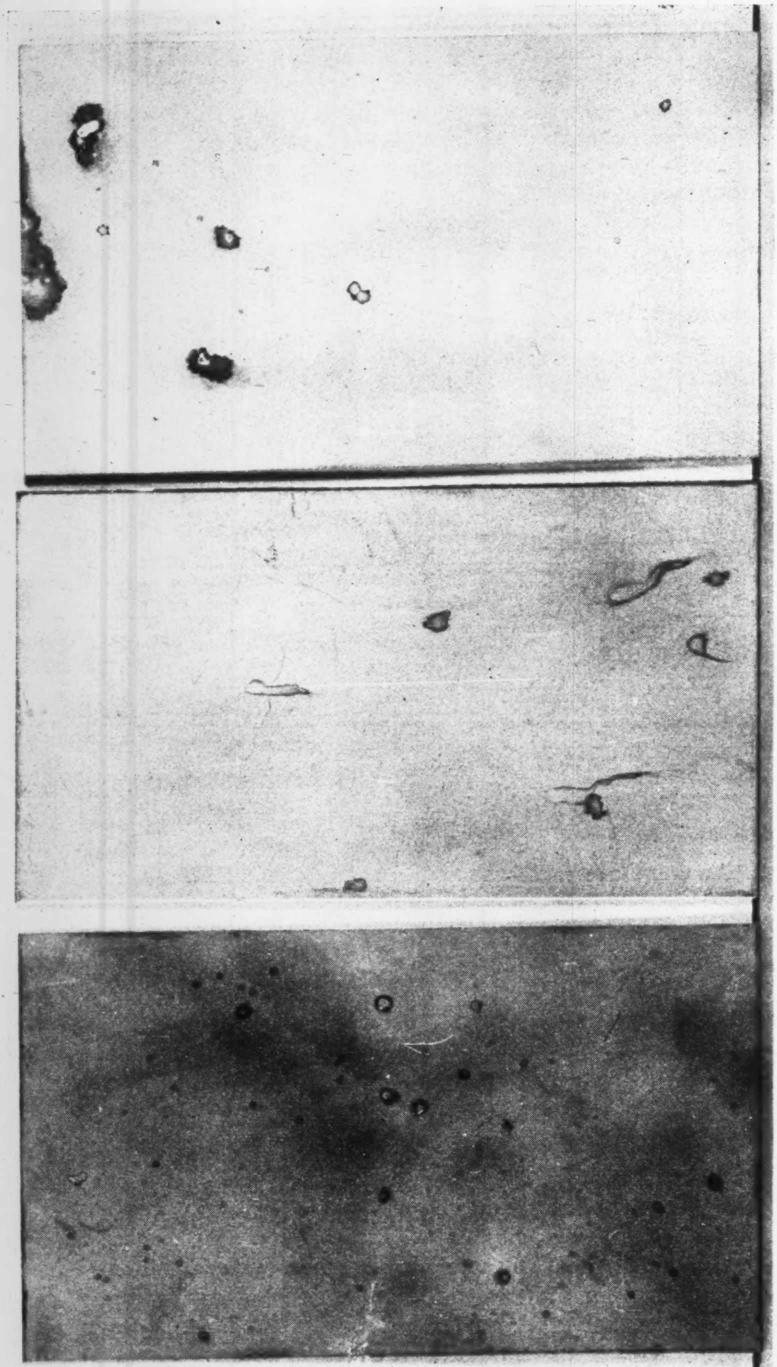
Heat	Composition, per cent					Boiling, Glacial-Acetic, in. per month		Sulfurous Acid, G Loss in 28 days		Boiling, 10 per cent Phosphoric Acid, in. per month	Boiling, 50 per cent Lactic Acid, in. per month
	Carbon	Manganese	Chromium	Nickel	Nitrogen	Liquid	Vapor	Liquid	Vapor		
DM32....	0.090	3.80	17.74	4.07	0.12	0.00015	0.0016[b]	Nil	Nil	Nil	...
DM35....	0.023	5.58	17.88	4.15	0.15	0.00014	0.0007[b]	Nil	0.0001	Nil	0.0340
DF76....	0.066	7.93	17.42	4.00	0.11	0.00078[a]	0.00030[a]	0.0002	0.0002[b]	Nil	0.0327
25311....	0.10	10.04	17.36	4.03	0.13	0.00021	0.0010	0.0002	0.0004[b]	Nil	0.0304
DQ47....	0.10	13.44	17.28	3.97	0.16	0.00029	...	Nil	Nil[b]	0.00019	...
ED56....	0.10	17.71	17.25	3.91	0.17	...	...	...	...	0.00028 0.00031	0.1131 ...

[a] Two periods.

[b] Pitting.

17 per cent chromium and 4 per cent nickel with manganese from 3.80 per cent to 17.71 per cent were included. Results show a very gradual increase in corrosion rates with increasing manganese content, slightly over $1\frac{1}{2}$ times from the low to the high end of the manganese range. It appears that higher corrosion rates found with the chromium-manganese steels with no nickel are probably due to the absence of nickel rather than the high manganese content.

In other corrosive media such as boiling glacial acetic acid, boiling 10 per cent phosphoric acid, and boiling 50 per cent lactic acid, manganese does not improve corrosion resistance nor harm it. The very low rates in 10 per cent phosphoric



17.91 Cr
4.27 Ni
6.22 Mn

17.36 Cr
4.03 Ni
10.04 Mn

17.27 Cr
3.91 Ni
17.71 Mn

FIG. 4.—Influence of Manganese on Salt-Spray Resistance.

acid even at 17.71 per cent manganese indicate a passive condition. These are shown in Table V.

Resistance to localized attack does not vary any extent over the manganese range covered. The photograph in Fig. 4 shows that a 17-per cent manganese heat behaves practically the same as the 6-per cent manganese heat. None of the three specimens shown are as resistant as type 304 to salt spray.

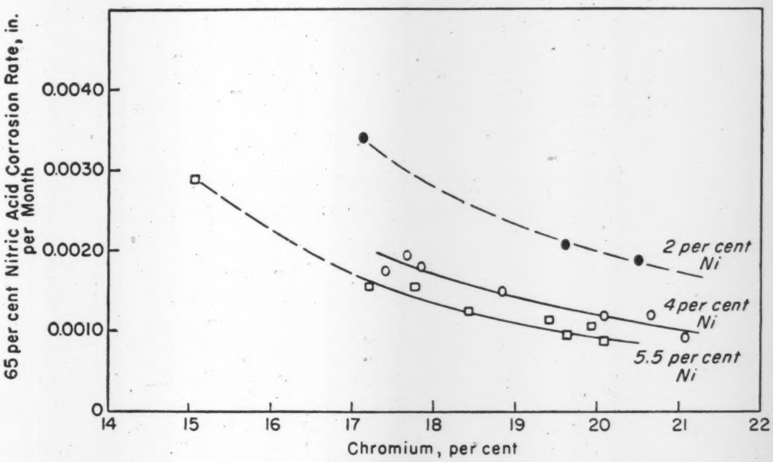


FIG. 5.—Influence of Chromium Content on Nitric-Acid Corrosion Rates of Chromium-Nickel-Manganese Steels at 2, 4, and 5.5 per cent Nickel.

Influence of Chromium:

Chromium would be expected to contribute largely to corrosion resistance of the chromium-nickel-manganese steels, especially under oxidizing conditions. This section deals with the influence of chromium and shows how its beneficial effect may or may not be vitiated by increasing manganese and decreasing nickel.

The results of the nitric acid tests are a very good indication of behavior under oxidizing conditions, where chromium has a pronounced significance. Figure 5 deals with the different analyses listed in Table VI. There are three different nickel levels shown, corresponding to a minimum of 2 per cent and a maximum of 5.5 per cent. This was done to determine whether nitric acid resistance comparable to 18-8 steel could be attained by increasing chromium much higher than it occurs in type 202. It is necessary to increase nitrogen content above 0.25 per cent if chromium is increased above 17 per cent, in order to maintain an austenitic structure. The data also show that nitrogen does not seriously harm nitric acid resistance in the annealed, solution-heat-treated conditions. Manganese is constant in all these compositions.

The results illustrate a definite improvement in corrosion resistance as the chromium content is raised, regardless of the nickel level. It can be seen that with only 2 per cent nickel it would be impossible to approach behavior of an 18-8 steel even with very high chromium content. A 20 chromium, 2 nickel, 10 manganese steel is equal or even slightly inferior to a type 202 steel. On the 5.5 per cent nickel curve it is apparent that behavior in nitric acid comparable to type 304 is readily attained at 20 per cent chromium. At the other end of this curve a 15 per cent chromium steel shows nitric

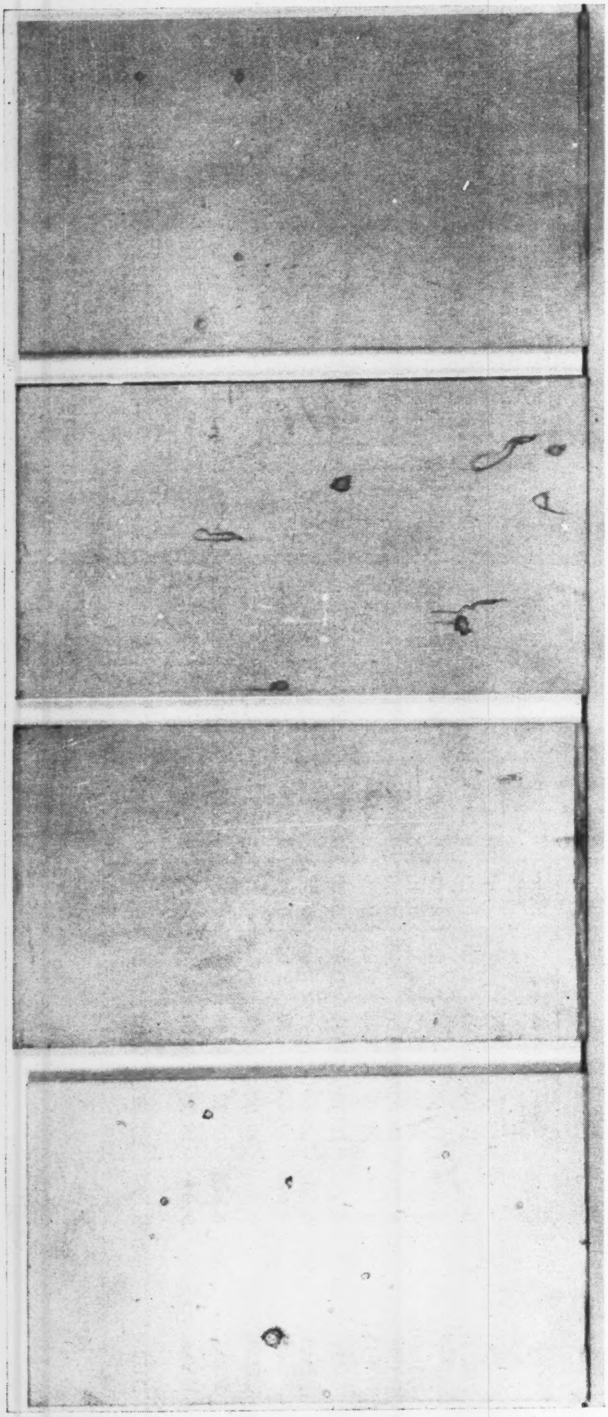
TABLE VI.—INFLUENCE OF CHROMIUM ON NITRIC ACID CORROSION RATES.

Heat	Carbon	Chromium	Manganese	Nickel	Nitrogen	Corrosion Rate, in. per month					
						1st	2nd	3rd	4th	5th	Average of five
25311	0.10	17.36	10.04	4.03	0.13	0.0019 0.0020	0.0017 0.0018	0.0016 0.0016	0.0017 0.0017	0.0017 0.0018	0.0017 0.0018
59518	0.12	17.69	10.35	4.08	0.15	0.0022 0.0023	0.0018 0.0019	0.0017 0.0017	0.0018 0.0018	0.0019 0.0019	0.0019 0.0019
DF77	0.056	17.83	9.89	4.06	0.13	0.0021 0.0018	0.0019 0.0021	0.0019 0.0018	0.0016 0.0016	0.0016 0.0016	0.0018 0.0018
DT100	0.098	18.82	10.02	4.11	0.40	0.0017 0.0017	0.0017 0.0017	0.0016 0.0016	0.0016 0.0014	0.0015 0.0012	0.0016 0.0015
DT99	0.10	20.05	10.26	4.18	0.39	0.0014 0.0013	0.0012 0.0012	0.0011 0.0012	0.0012 0.0012	0.0011 0.0011	0.0012 0.0012
DU7	0.11	20.66	9.85	4.14	0.42	0.0016 0.0016	0.0013 0.0013	0.0011 0.0011	0.0011 0.0010	0.0010 0.00096	0.0012 0.0012
DW16	0.030	21.02	10.22	4.01	0.36	0.0011 0.0010	0.0010 0.00097	0.00094 0.00090	0.00089 0.00086	0.00085 0.00080	0.00096 0.00091
ED40	0.11	17.13	10.17	1.97	0.20	0.0038 0.0036	0.0034 0.0035	0.0035 0.0035	0.0032 0.0031	0.0029 0.0029	0.0034 0.0033
ED42	0.10	19.60	10.17	1.99	0.31	0.0022 0.0023	0.0021 0.0022	0.0020 0.0021	0.0019 0.0020	0.0020 0.0020	0.0020 0.0021
ED45	0.090	20.52	10.28	2.04	0.31	0.0019 0.0019	0.0018 0.0018	0.0019 0.0019	0.0019 0.0019	0.0018 0.0018	0.0019 0.0019
ED55	0.10	15.02	8.66	5.94	0.21	0.0032 0.0032	0.0028 0.0028	0.0030 0.0030	0.0028 0.0028	0.0029 0.0029	0.0029 0.0029
25350	0.024	17.22	9.99	5.39	0.20	0.0016 0.0017	0.0016 0.0016	0.0016 0.0016	0.0015 0.0016	0.0017 0.0016	0.0016 0.0016
18007	0.089	17.75	9.14	5.28	0.14	0.0016 0.0016	0.0017 0.0017	0.0017 0.0017	0.0015 0.0016	0.0016 0.0016	0.0016 0.0016
DY35	0.020	18.37	9.09	5.45	0.20	0.0013 0.0012	0.0012 0.0012	0.0012 0.0011	0.0013 0.0012	0.0013 0.0013	0.0013 0.0012
DU10	0.032	19.37	9.16	5.50	0.40	0.0014 0.0014	0.0011 0.0011	0.0011 0.0011	0.0011 0.0011	0.0011 0.0011	0.0012 0.0012
DY34	0.019	19.60	9.07	5.49	0.20	0.0011 0.0010	0.00089 0.00090	0.00091 0.00095	0.00091 0.00094	0.00082 0.00084	0.00091 0.00092
DU17	0.095	19.97	9.32	5.64	0.40	0.0013 0.0012	0.0011 0.0012	0.00098 0.0010	0.0011 0.0011	0.0010 0.0010	0.0011 0.0011
DU21	0.030	20.01	9.00	5.50	0.32	0.0011 0.0010	0.00080 0.00085	0.00084 0.00083	0.00081 0.00080	0.00082 0.00083	0.00087 0.00087

TABLE VII.—INFLUENCE OF CHROMIUM ON CORROSION RATES UNDER INTERMEDIATE CONDITIONS AND IN ORGANIC MEDIA.

Heat	Composition, per cent					Boiling, Glacial-Acetic, in. per month		Sulfurous Acid, G Loss per 28 days		Boiling, 10 per cent Phosphoric Acid, in. per month	10 per cent Citric Acid, 200 F, in. per month	Boiling, 50 per cent Lactic Acid, in. per month	10 per cent Oxalic Acid, 200 F, in. per month
	Carbon	Chromium	Manganese	Nickel	Nitrogen	Liquid	Vapor	Liquid	Vapor				
25311	0.10	17.36	10.04	4.03	0.13	0.00021	0.0010	0.0002	0.0004[a]	Nil	0.00026 0.00022	0.0304 ...	0.0243 0.0227
DF77	0.056	17.83	9.89	4.06	0.13	0.00024	0.0013	0.0004	0.0005	Nil	...	...	...
DT100	0.098	18.82	10.02	4.11	0.40	0.00025	...	Nil	0.0004	Nil	...	...	...
DT99	0.10	20.05	10.26	4.18	0.39	0.00028	...	Nil	Nil	0.0005	...	...	...
DW16	0.030	21.02	10.22	4.01	0.36	0.00004	...	Nil	0.0020[a]	Nil	...	...	...
ED40	0.11	17.13	10.17	1.97	0.20	0.00005	0.00095	...	...	0.00045 0.00034	0.00056 ...	0.0460 ...	... ...
ED42	0.10	19.60	10.17	1.99	0.31	...	...	0.0007	0.0016[a]	Nil	...	Nil	...
ED45	0.090	20.52	10.28	2.04	0.31	0.00009	...	0.0004	0.0011	Nil	...	Nil	...
ED55	0.10	15.02	8.66	5.94	0.21	...	...	...	...	0.0863 0.0849	0.0060 ...	0.0792 ...	0.0206 0.0258
25350	0.024	17.22	9.99	5.39	0.20	0.00024	0.0010	0.0003	0.0005[a]	Nil	...	...	...
DY35	0.020	18.37	9.09	5.45	0.20	0.00003	...	Nil	Nil	Nil	... ...	... ...	0.0282 0.0282
DU21	0.030	20.01	9.00	5.50	0.32	0.00002	...	0.0005	0.0007	Nil	0.00017 0.00022	0.00013 0.00016	0.0271 0.0284

[a] Pitting.



15.02 Cr
5.94 Ni
8.66 Mn

19.60 Cr
5.49 Ni
9.07 Mn

17.36 Cr
4.03 Ni
10.04 Mn

20.66 Cr
4.14 Ni
9.85 Mn

FIG. 6.—Influence of Chromium on Salt-Spray Resistance.

acid loss only 3 to 4 times that of type 304. These are as good as the best type 430 and superior to average type 430. These data lead to the suggested analysis of 20 per cent chromium-6 per cent nickel-8 per cent manganese as an optimum composition with corrosion and mechanical properties like those of type 304. The advantages of this composition for other corrosive conditions are illustrated in Table VII.

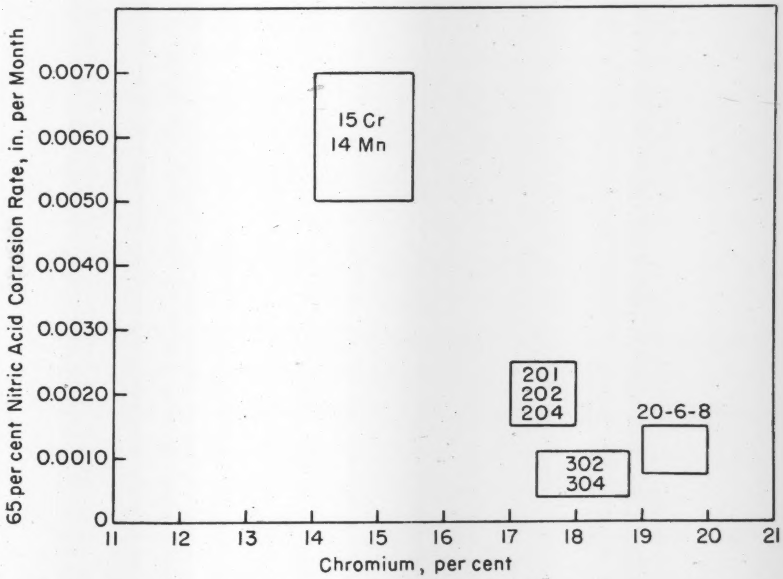


FIG. 7.—Comparative Nitric Acid Resistance of Chromium-Manganese, Chromium-Nickel-Manganese, and Chromium-Nickel Steels.

The data in Table VII show no marked effect of chromium between 17 per cent and 20 per cent under intermediate conditions in the acetic acid and sulfurous acid test. When chromium contents fall below 17 per cent, results show increased rates of attack in acetic acid (refer to heat ED36, Table III). Further, the 15 per cent chromium (ED55) steel is attacked by boiling 10 per cent phosphoric acid and 10 per cent citric acid at 200 F, both rather weak corrosive media.

In the stronger organic acids, such as boiling 50 per cent lactic, three chromium-nickel-manganese steels offer good resistance. All three of these contain 19.6 per cent or more chromium. Indications are that performance in this instance is governed by chromium content, and these high-chromium-chromium-nickel-manganese steels excel type 304 for this reason. However, even 20 per cent chromium is not sufficient to obtain satisfactory resistance to hot oxalic acid, but this still does not represent inferiority to type 304 which also is susceptible to attack under these conditions.

The superior properties of 20 per cent chromium-6 per cent nickel-10 per cent manganese steels in environments which promote localized attack are shown in salt spray tests pictured in Fig. 6. It can readily be noted that increasing amounts of chromium improve the salt-spray resistance. However, it does not have sufficient ability to accomplish this without the aid of nickel. While improvement over a type 202 is realized with a 20

TABLE VIII.—COMPARISON OF CHROMIUM-NICKEL-MANGANESE STEELS WITH TYPE 304 AND CHROMIUM-MANGANESE STEEL.

Class	Heat	Composition, per cent					Nitric Acid,[a] ipm.	Boiling, Glacial-Acetic, in. per month		Sulfurous Acid, G Loss per 28 days		Boiling, 10 per cent Phosphoric Acid, in. per month	10 per cent Oxalic, 200 F., in. per month	Boiling, 50 per cent Lactic, in. per month
		Carbon	Chromium	Manganese	Nickel	Nitrogen		Liquid	Vapor	Liquid	Vapor			
15-14-1........	CW98........	0.07	16.32	15.44	0.87	0.13	0.0047 0.0048	0.0021	0.0020	0.0647	0.0121	...	0.0358	0.124
	88884........	0.11	15.96	15.15	1.11	0.14	0.0048 0.0051	0.0081	0.0029	...	...	...	0.0217	...
	23323........	0.094	15.44	16.89	0.99	0.27	0.0067 0.0069	...	...	...	...	0.0534	...	...
201...........	25367........	0.11	17.91	6.22	4.27	0.14	0.0016 0.0016	0.00025	0.0004	0.0003	0.0004	Nil	0.0249 0.0236	0.0379 0.0367
	25312........	0.097	16.76	5.90	3.81	0.14	0.0020 0.0021	...	...	...	...	Nil	...	...
202...........	18007........	0.087	17.75	9.14	5.28	0.14	0.0013 0.0014	...	...	...	...	Nil	...	...
	25311........	0.10	17.36	10.04	4.03	0.13	0.0017 0.0018	0.00021	0.0010	0.0002	0.0004	Nil	0.0243 0.0227	0.0329 0.0304
20-6-S.........	DU7.........	0.11	20.66	9.85	4.14	0.42	0.0012 0.0012	0.00007	...	0.0009	0.0005	...	...	...
	DU21........	0.030	20.01	9.00	5.50	0.32	0.00087 0.00087	0.00002	...	0.0005	0.0007	Nil	0.0271 0.0284	0.00013 0.00016
304...........	98963........	0.044	18.24	1.64	9.56		0.00087 0.00085	0.00011	0.0004	0.0004	0.0007	Nil	0.0065 0.0065	0.0804 0.0976

---

[a] Average of five periods.

chromium, 4 nickel, 10 manganese steel, this higher chromium analysis is still less resistant than type 304. With 6 per cent nickel, the 20 chromium, 6 nickel, 8 manganese steel shows excellent salt-spray resistance.

Based on these data, a 20-6-8 steel would probably be suitable for any severe applications presently handled by type 304.

COMPARISON OF EXISTING CHROMIUM-MANGANESE, CHROMIUM - NICKEL - MANGANESE AND CHROMIUM-NICKEL GRADES

Although the influence of the three elements—chromium, nickel, and manganese—have been discussed, such information can be made more useful by comparing the standard types 201, 202, and 304 with a straight chromium, manganese steel and with the special 20-6-8 analysis. This will establish the position of chromium-manganese steels, show why nickel is essential, and indicate what can be done to equal type 304 for severe environments.

TABLE IX.—COMPOSITION OF 17-4-10 CHROMIUM, NICKEL, MANGANESE HEATS TESTED FOR INTERGRANULAR CORROSION IN TABLE X AND FIG. 8.

Heat	Carbon	Manganese	Silicon	Chromium	Nickel	Nitrogen
DT87-3	0.017	10.13	0.28	17.36	4.0	0.26
25350	0.023	10.35	0.56	17.36	4.97	0.20
DG64	0.040	08.33	0.47	18.19	4.08	0.15
DT78A	0.050	09.96	0.27	17.86	3.92	0.22
DF76	0.066	07.93	0.14	17.42	4.00	0.11
DT78B	0.069	09.90	0.30	17.72	3.91	0.21
DT78C	0.090	09.86	0.26	17.63	3.97	0.21

Figure 7 represents the relative positions of these various steels based on nitric acid corrosion tests. This particularly illustrates the advantage of steels containing 4 per cent or more nickel, with chromium content of 17 per cent or more. These steels, which are now standard types 201 and 202 also possess mechanical properties equivalent to their respective chromium-nickel counterparts types 301 and 302.

Table VIII covers a summary of corrosion tests in nitric acid as well as other media. These indicate that a type 201 or 202 would probably be satisfactory for all applications now handled by Types 301 and 302. It should be mentioned here that the 15 chromium-14 manganese-1 nickel steels are suitable for certain intermediate conditions, such as those where type 430 has been used.

For conditions involving welding or extreme corrosive environments where type 304 is now employed, two alternatives may be used: Types 204 or 204L with 0.10 per cent maximum carbon and 0.03 per cent maximum carbon respectively would be suitable where welding is required and the 20-6-8 analysis should complete the picture where superior corrosion resistance is needed. It equals type 304 in boiling nitric acid and the salt-spray test and even excels type 304 in boiling 50 per cent lactic acid. This improved 20-6-8 composition also possesses mechanical properties the same as type 304. Its use would still enable a saving of 3 per cent or 4 per cent nickel.

INTERGRANULAR CORROSION

The chromium - nickel - manganese steels, similar to the plain chromium, nickel steels, are subject to intergranular corrosion if heated in the 1000 to 1600 F temperature range. Since carbon is the controlling factor for this phenomenon, a group of heats with increasing carbon content from 0.017 to 0.10 per cent shown in Table IX were sensitized for various lengths of time at 1200 F and then tested in the standard 65 per cent boiling nitric acid and acidified copper sulfate tests. The results are tabulated in Table X and plotted in Fig. 8 as corrosion losses *versus* sensitizing time. A short sensitizing time of 45 min at 1200 F cannot produce intergranular corrosion in any of the heats tested. Two hours sensitizing does not produce intergranular corrosion in the heats up to and including 0.050 per cent carbon. The other heats with 0.066 per cent and higher carbon content failed in both tests. A 24- and 100-hr sensitizing time produced intergranular corrosion in all heats, except DT87 and 25350 with 0.017 per cent and 0.023 per cent carbon respectively, and even these failed in the nitric acid test but passed the copper sulfate test in the same manner as types 304L and 347. It appears from these results that the behavior of the chromium-nickel-manganese steels of nominal composition 17 chromium, 4 nickel, 10 manganese from the intergranular corrosion standpoint are very similar to the regular chromium-nickel grades. Lowering the carbon content prevents intergranular corrosion

TABLE X.—RESULTS OF COPPER SULFATE AND BOILING NITRIC ACID TESTS ON CHROMIUM-NICKEL-MANGANESE STEELS SENSITIZED AT 1200 F.

Heat	Heat Treatment	Boiling 65 per cent Nitric Test, Period, in. per month[a]						Copper Sulfate Test
		1st	2nd	3rd	4th	5th	Average of five	
DT87-3....	As Annealed........	0.0017	0.0016	0.0017	0.0016	0.0016	0.0016	Passed
	1200 F—45 min.....	0.0016	0.0017	0.0016	0.0017	0.0016	0.0016	Passed
	1200 F—2 hr.......	0.0017	0.0017	0.0016	0.0017	0.0016	0.0017	Passed
	1200 F—24 hr......	0.0021	0.0068	0.0075	...	...	...	Passed
	1200 F—100 hr.....	0.0030	0.0095	...	...	...	...	Passed
25350......	As Annealed........	0.0016	0.0016	0.0016	0.0015	0.0017	0.0016	Passed
	1200 F—1 hr.......	0.0015	0.0017	0.0018	0.0018	0.0020	0.0018	Passed
	1200 F—2 hr.......	0.0016	0.0018	0.0021	0.0022	0.0024	0.0020	Passed
	1200 F—24 hr......	0.0033	0.0077	...	...	...	...	Passed
DG64......	As Annealed........	0.0020	0.0018	0.0016	0.0014	0.0015	0.0017	Passed
	1200 F—2 hr.......	0.0022	0.0018	0.0017	0.0018	0.0021	0.0019	Passed
	1200 F—24 hr......	0.0032	0.0057	0.0076	0.0108	0.0135	0.0081	Failed
DT78A....	As Annealed........	0.0017	0.0016	0.0017	0.0016	0.0016	0.0017	Passed
	1200 F—45 min.....	0.0017	0.0016	0.0015	0.0015	0.0014	0.0015	Passed
	1200 F—2 hr.......	0.0019	0.0020	0.0021	0.0024	0.0027	0.0022	Passed
	1200 F—24 hr......	0.011	...	...	...	...	...	Failed
	1200 F—100 hr.....	0.040	...	...	...	...	...	Disintegrated
DF76......	As Annealed........	0.0018	0.0017	0.0016	0.0016	0.0015	0.0016	Passed
	1200 F—2 hr.......	0.0015	0.0020	0.0025	0.0034	0.0041	0.0027	Failed
	1200 F—24 hr......	0.0041	0.0065	0.0093	0.0153	0.0248	0.0120	Failed
DT78B....	As Annealed........	0.0019	0.0018	0.0017	0.0017	0.0017	0.0017	Passed
	1200 F—45 min.....	0.0019	0.0019	0.0019	0.0020	0.0021	0.0020	Passed
	1200 F—2 hr.......	0.0021	0.0024	0.0033	0.0105	...	...	Failed
	1200 F—24 hr......	0.0288	...	...	...	...	...	Disintegrated
	1200 F—100 hr.....	0.0447	...	...	...	...	...	Disintegrated
DT78C....	As Annealed........	0.0018	0.0018	0.0018	0.0018	0.0018	0.0018	Passed
	1200 F—45 min.....	0.0020	0.0025	0.0032	0.0039	0.0040	0.0031	Passed
	1200 F—2 hr.......	0.0028	0.0043	0.0059	0.0175	...	...	Disintegrated
	1200 F—24 hr......	0.0355	...	...	...	...	...	Disintegrated
	1200 F—100 hr.....	0.056	...	...	...	...	...	Disintegrated

[a] Average of duplicate specimens.

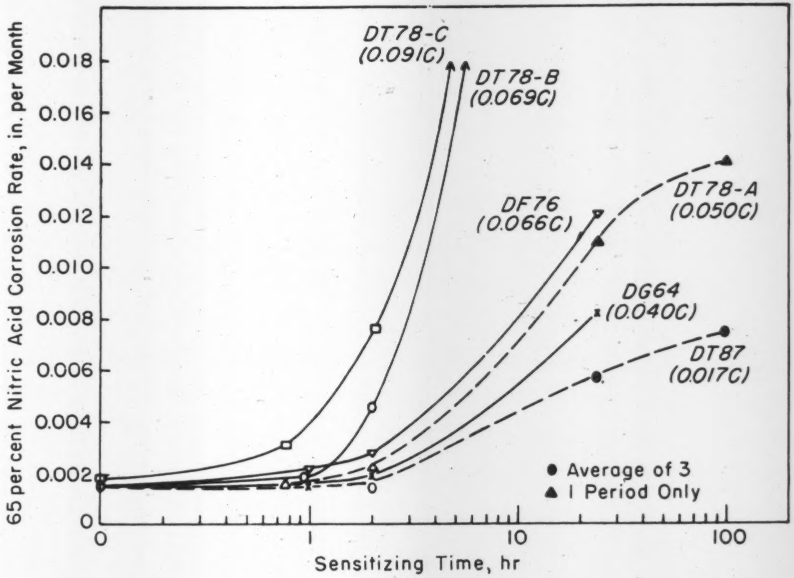


FIG. 8.—Effect of Sensitizing Time at 1200 F on Nitric Acid Losses of 17-4-10 Chromium-Nickel-Manganese Steel With Varying Carbon Contents.

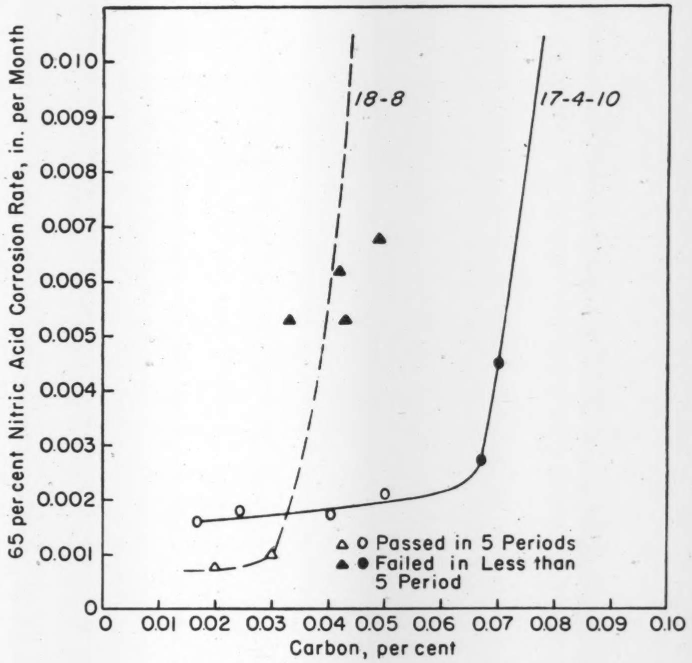


FIG. 9.—Comparison of 17-4-10 Chromium-Nickel-Manganese With 18-8 Chromium-Nickel Steel in 65 per cent Nitric-Acid Test, Samples Sensitized 2 hr at 1200 F.

but only in shorter sensitizing time. The low-carbon chromium-nickel-manganese steels can pass these corrosion tests after sensitizing 1 hr at 1200 F. In 24-hr sensitizing time, even the low-carbon composition shows intergranular corrosion, as is the case with types 304L and 347.

TABLE XI.—COMPOSITION OF 20-6-8 CHROMIUM, NICKEL, MANGANESE HEATS TESTED FOR INTERGRANULAR CORROSION IN TABLE XII.

Heat	Carbon	Silicon	Manganese	Chromium	Nickel	Nitrogen
DU21........	0.030	0.41	9.00	20.01	5.50	0.20
DU10........	0.032	0.52	9.16	19.37	5.50	0.40
DW39.......	0.11	0.39	9.45	18.55	5.46	0.32

TABLE XII.—RESULTS OF BOILING NITRIC-ACID AND COPPER-SULFATE TESTS FOR 20-6-8 CHROMIUM-NICKEL-MANGANESE HEATS SENSITIZED AT 1200 F.

Heat	Heat Treatment	Boiling 65 per cent Nitric Test Period, in. per month[a]						Copper Sulfate Test
		1st	2nd	3rd	4th	5th	Average of five	
DU10......	As Annealed.........	0.0014	0.0011	0.0011	0.0011	0.0011	0.0012	Passed
	1200 F—1 hr........	0.0017	0.0017	0.0017	0.0016	0.0017	0.0017	Passed
	1200 F—2 hr........	0.0020	0.0021	0.0023	0.0024	0.0024	0.0022	Passed
	1200 F—24 hr.......	0.0071	...	...	...	...	...	Failed
	1200 F—100 hr......	0.0077	...	...	...	...	...	Failed
DU21......	As Annealed.........	0.0011	0.00080	0.00084	0.00081	0.00082	0.00087	Passed
	1200 F—1 hr........	0.0011	0.0011	0.00080	0.00086	0.00086	0.00093	Passed
	1200 F—2 hr........	0.0012	0.0011	0.00087	0.00090	0.00092	0.0010	Passed
	1200 F—24 hr.......	0.0019	0.0063	...	...	...	...	Failed
	1200 F—100 hr......	0.0024	0.0075	...	...	...	...	Failed
DW39.....	As Annealed.........	0.0013	0.0011	0.0010	0.0012	0.0015	0.0012	Passed
	1200 F—1 hr........	0.0027	0.0454	...	...	...	...	Failed

[a] Average of duplicate specimens.

The carbon content seems to be less critical in the chromium-nickel-manganese steels than in the chromium-nickel grades, as can be seen in Fig. 9 where the corrosion losses in 65 per cent nitric acid have been plotted *versus* carbon content for chromium-nickel-manganese as well as chromium-nickel steels. The data for chromium-nickel steels were obtained in the Allegheny Ludlum Research Laboratory on commercial 304L. The sensitizing time used was 2 hr at 1200 F. From this figure it seems that the maximum carbon content of the chromium-nickel-manganese steels corresponding to the 0.03 per cent maximum in chromium-nickel steels is between 0.05 and 0.06 per cent.

In order to investigate the intergranular corrosion resistance of the higher chromium and nickel alloys of the nominal composition designated 20-6-8, the heats shown in Table XI were sensitized and tested in 65 per cent boiling nitric and in acidified copper sulfate. The results are given in Table XII. Heats DU10 and DU21 with 0.030 and 0.032 per cent carbon respectively are immune to intergranular corrosion after being sensitized 2 hr at 1200 F but fail in both tests after a 24-hr sensitizing time. Heat DW39 with 0.011 per cent carbon is susceptible to intergranular corrosion after 1 hr at 1200 F and it was not found necessary to lengthen the sensitizing time. It seems, therefore, that sensitized 20-6-8 chromium-nickel-manganese steels, behave in the same way as the 17-4-10 chromium-nickel-manganese steels, although no attempt was made to find whether the critical carbon content is the same.

All the sensitized samples have been examined microscopically. Heat DT87 with 0.017 per cent carbon and 0.26 per cent nitrogen is free of grain boundary precipitate even after 100 hr at 1200 F as shown in Fig. 10. Grain boundary precipitation is found in higher carbon material. Heat DG64 with 0.040 per cent carbon and 0.15 per cent nitrogen shows no carbide precipitation in 2 hr, but in 24 hr a discontinuous grain boundary precipitate forms. In the other heats with higher carbon content grain boundary precipitation occurs more readily. The photomicrograph in Fig. 11 shows grain boundary precipitation in heat DT78C sensitized 1 hr at 1200 F. In general, the morphology of carbide precipitation in chromium-nickel-manganese steel is the same as in chromium-nickel of equivalent carbon content in spite of the presence of higher nitrogen content.

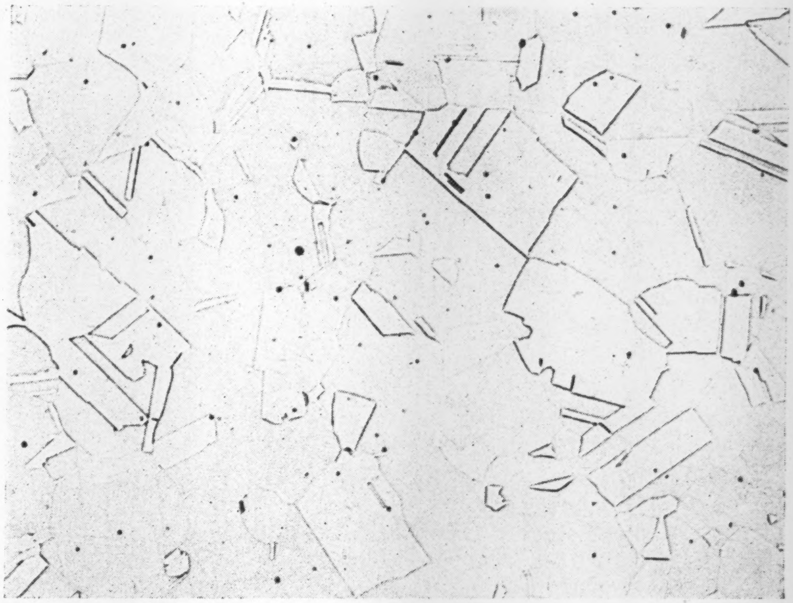


FIG. 10.—Heat DT87 (0.017 per cent Carbon and 0.26 per cent Nitrogen) Sensitized 100 hr at 1200 F. No Grain Boundary Precipitation. Etched in NaCN (×250).

STRESS CORROSION

All of the chromium-nickel-manganese steels with an austenitic structure are subject to stress-corrosion cracking in the boiling 42 per cent magnesium-chloride test. This test is very severe and such conditions are probably never met in service, but it does offer a good way of determining compositions which may be more resistant than others to this type of localized corrosion. The results have not been tabulated because all specimens cracked in times varying from 4 hr to 72 hr, and no influence of chromium, nickel, or manganese could be associated with these. The only compositions which showed no cracking were those which contained delta ferrite, and these remain free of cracks up to 250 hr.

These data indicate in general that the chromium-nickel-manganese steels probably would perform about the same as chromium-nickel steels under conditions which are conducive to stress corrosion.

SUMMARY AND CONCLUSIONS

The corrosion properties of chromium-nickel-manganese austenitic steels have been evaluated in various media and the influence of individual elements has been established.

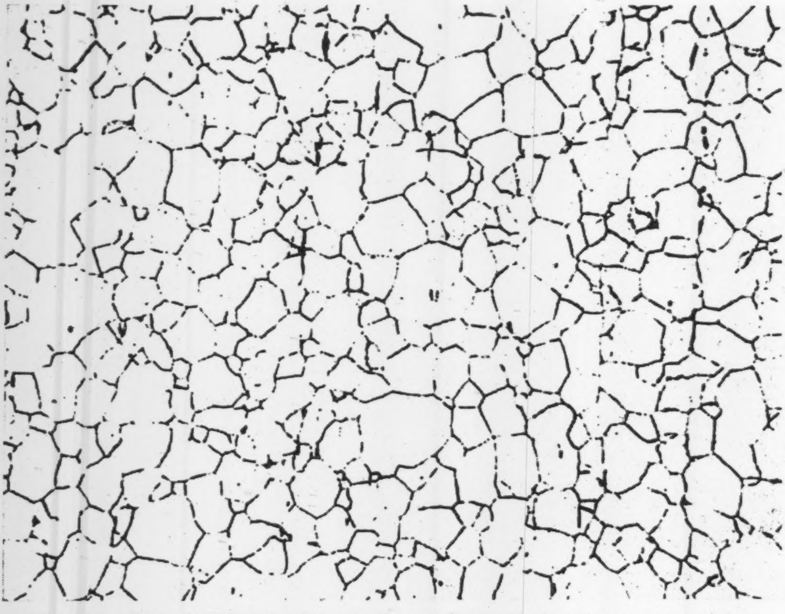


FIG. 11.—Heat DT87C Sensitized 1 hr at 1200 F. Etched in NaCN (×250)

The following composition range was investigated: chromium, 15 to 21 per cent; nickel, 2 to 6.50 per cent; manganese, 5 to 17 per cent.

The influence of nickel is prominent in these chromium-nickel-manganese steels. In boiling nitric acid tests, at 17 per cent chromium level the improvement due to increasing nickel is more pronounced than at a 20 per cent chromium level. Nitric acid corrosion rates comparable to 18-8 can be attained with the 20 per cent chromium, 6 per cent nickel, manganese steel, and this composition also shows very good resistance, as does 18-8, to localized attack and to boiling 50 per cent lactic acid.

Manganese can be varied from 4 per cent to 14 per cent with no significant effect on corrosion resistance. On the other hand chromium produces considerable improvement as it is increased, based on boiling nitric acid data, certain organic acids and salt-spray results.

The types 201 and 202 are equal to types 301 and 302 in many mild environments like acetic, citric and phosphoric acids. For more severe conditions indicated by the boiling nitric test or for localized conditions, a 20 per cent chromium, 6 per cent nickel, 8 per cent manganese composition has been developed which is comparable to type 304.

The intergranular corrosion resistance of the chromium-nickel-manganese steels is the same as that of chromium-nickel steels. Lowering the carbon content prevents intergranular corrosion but only in short sensitizing times, similar to type 304L. The carbon content seems, however, to be less critical than in the chromium-nickel grades and it seems that the maximum carbon content corresponding to the 0.03 per cent maximum in chromium-nickel steels is between 0.05 and 0.06 per cent for the chromium-nickel-manganese steels.

The chromium - nickel - manganese steels are subject to stress corrosion cracking like the chromium-nickel steels. Resistance to stress corrosion does not appear to have any association with the amount of chromium, nickel, or manganese provided the structure is austenitic. The presence of delta ferrite can prevent or decrease susceptibility to such failure.

DISCUSSION

Mr. R. W. Manuel.[1]—The data on what we would call low-temperature corrosion is very interesting and very useful, but in the petroleum industry we have a particularly difficult problem in finding materials to resist high-temperature corrosion in hydrogen atmospheres in catalytic reforming and desulfurization and cracking processes in the presence of hydrogen sulfide. The chromium-nickel austenitic stainless steels are being used for severe conditions, and we wonder if the authors are doing any work at all on the manganese steels.

There is some German work which indicates that manganese might be useful in improving resistance to high-temperature hydrogen sulfide attack.

Mr. Roland Wardell.[2]—We have made some preliminary investigations of the manganese-bearing stainless steel in 50 per cent sulfuric acid, and we find that it was slightly superior to the comparable 18-8 stainless steels. Have the authors made any investigations into the use of manganese-bearing stainless steel in sulfuric acid?

Mr. W. G. Renshaw (*author*).—In answer to Mr. Manuel's question, we have not as yet carried out any investigation of the resistance of these chromium-nickel-manganese steels to hydrogen sulfide. Meager data so far do not indicate them to be any advantage over the chromium-nickel austenitic steels.

In reply to Mr. Wardell, we have carried out corrosion tests in sulfuric acid. What per cent concentration is involved?

Mr. Wardell.—Fifty per cent.

Mr. Renshaw.—Was this at an elevated temperature and did rather appreciable attack of the material occur?

Mr. Wardell.—Yes.

Mr. Renshaw.—We have not carried out sulfuric acid tests to determine corrosion rates in this range. Most of our interest has been only in those ranges where the stainless steels are passive. However, both the Cr-Ni-Mn and the Cr-Ni steels are active under these conditions. While calculated corrosion rates at high temperatures and fifty per cent sulfuric acid may be slightly less for chromium-nickel-manganese steels than 18-8, nevertheless both show such high rates that the difference is of little significance. Have corrosion rates been determined?

Mr. Wardell.—No, they have not.

Mr. Renshaw.—Where corrosion rates are very low, say less than 0.0005 in. penetration per month, we can reasonably assume the metal is passive. Above this point indicates the metal is probably active and then corrosion rates for several specimens may vary over a rather broad range at any certain specific test condition. For this reason we have made no attempt to cite specific rates at these aggressive sulfuric acid concentrations.

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[1] Corrosion Engineer, Socony Mobil Oil Co., New York, N. Y.

[2] Laboratory Supervisor, Minneapolis Honeywell, Minneapolis, Minn.

FRETTING WEAR OF ZIRCALOY-2 PELLETS IN HIGH-TEMPERATURE WATER*[1]

By Louis A. Waldman[2] and Paul Cohen[2]

Synopsis

Pellet bed reactors using Zircaloy-2 cladding are attractive for pressurized-water reactors, because of the potential simplicity of the pellet bed reactor core and the low cross-section for neutron absorption of the zirconium. In such a reactor, fretting of adjacent pellets might be a problem because of the release of induced zirconium activity, and if wear is appreciable, perforation of the cladding. The paper describes an investigation at fully simulated operating conditions, in which frettting wear rates were determined by using irradiated pellets. The results of this test were combined with observations from previous tests and an analysis made of the hazards of this type of reactor. It is shown that neither activity release nor cladding penetration is seriously affected by fretting wear.

The use of the pellet type fuel element in pressurized-water reactor cores has been contemplated for various reasons. Among these are improvements in the pressure vessel design and in reactor operation due to the simplification of core structural supports and the ease of loading and unloading of the fuel material through fluidization.

Included among the unknown operating characteristics of a bed of Zircaloy-clad uranium dioxide pellets is the amount of fretting wear that might occur among the randomly packed pellets. Adjacent pellets that undergo some movement, microscopic or macroscopic, due to the action of the coolant could conceivably wear at points of contact with subsequent reduction in clad thickness, release of significant amounts of Zr^{95} activity into the coolant stream, and possibly ultimate perforation of the cladding.

To evaluate this problem, seven tests were made in the period September 1953 to June 1954, through the combined efforts of the Materials Department and Chemistry Subdivision, in a Bettis loop facility ("Bettis Loop M-1"). Pellet configurations consisting of spheres, dumbells, and els were tested with both upflow and downflow of water through beds at a velocity sufficient to fluidize the bed in upflow. The results of these tests indicated the general stability of the cladding material for the short-term tests conducted but were inconclusive as to rates of fretting wear because of the lack of sensitivity afforded by weight change measurements. Evidence of localized fretting wear was observed on the surfaces of a majority of the pellets tested, but the amount of fretting debris and depth of penetration at the fretting sites could not be determined.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] The opinions expressed herein are the authors only; and this report does not constitute an expression of official opinion of the Bettis Plant.

[2] Supervising Engineer, Central Technical Activities, and Manager, PWR Chemistry Section, respectively, Westinghouse Electric Corporation, Bettis Plant, Pittsburgh, Pa.

Subsequently, an analysis was made of significant fretting wear rates in order to evaluate the pellet fretting problem with respect to operating hazards associated with the contemplated reactor designs. The results of this analysis demonstrated the feasibility of a test with an irradiated bed of pellets yielding sufficient activity to trace the fretting wear rates of significance and compatible with safe handling procedures in the Bettis loop facilities, given a properly designed test section. Such a test was planned and carried out between July 1954 and Jan. 1955.

The results and conclusions obtained from the irradiated-pellet fretting test and their correlation with the previous seven tests are the bases for this report.

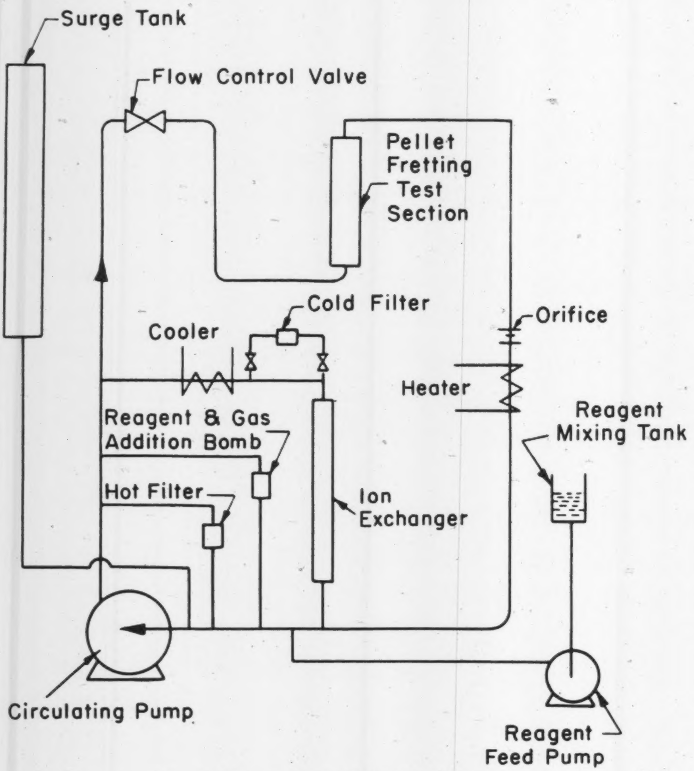
Surge Tank
Flow Control Valve
Pellet Fretting Test Section
Cold Filter
Cooler
Orifice
Heater
Reagent Mixing Tank
Reagent & Gas Addition Bomb
Hot Filter
Ion Exchanger
Circulating Pump
Reagent Feed Pump

FIG. 1.—Bettis Loop Facility Schematic.

EXPERIMENT AND PROCEDURE

Test Facility.—A Bettis loop facility ("Bettis Loop R-3") was modified for the accommodation of a test section compatible with the testing of a high-level activity source consisting of a bed of irradiated Zircaloy-2 pellets with a nominal total activity of five curies.[3] A schematic of the loop facility is shown in Fig. 1 and a photograph of a typical Bettis loop facility, in Fig. 2. The test section is shown in Fig. 3. Details of the can insert containing the bed of irradiated pellets are shown in Fig. 4. The test section consisted of a section of 3-in., Schedule-80 stainless steel pipe with a flanged top for insertion of the can and a holding pipe at the bottom for retention of the can insert within the loop. Lead bricks were stacked on the outside of the test section for shielding.

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[3] The activity of the bed as received was approximately 1 curie, apparently due to irradiation fluxes lower than those specified.

The can was loaded against a flexitallic gasket by means of a spring designed for a 100 lb load. This loading (more than a factor of two above the uplift on the bed resulting from its pressure drop) was utilized in order to minimize leakage across the holding ring. The load was transmitted through a combination of sliding couplings on the can connector rod and upper shielding plug. Since the pressure drop observed for the given run was equal to that observed for previous tests conducted on beds of approximately the same dimensions, it is reasonable to conclude that little or no leakage occurred across the interface between the can and the holding ring. The basic design described above was utilized to facilitate loading and unloading of the integral unit containing the bed through a suitable cask suspended over the test section.

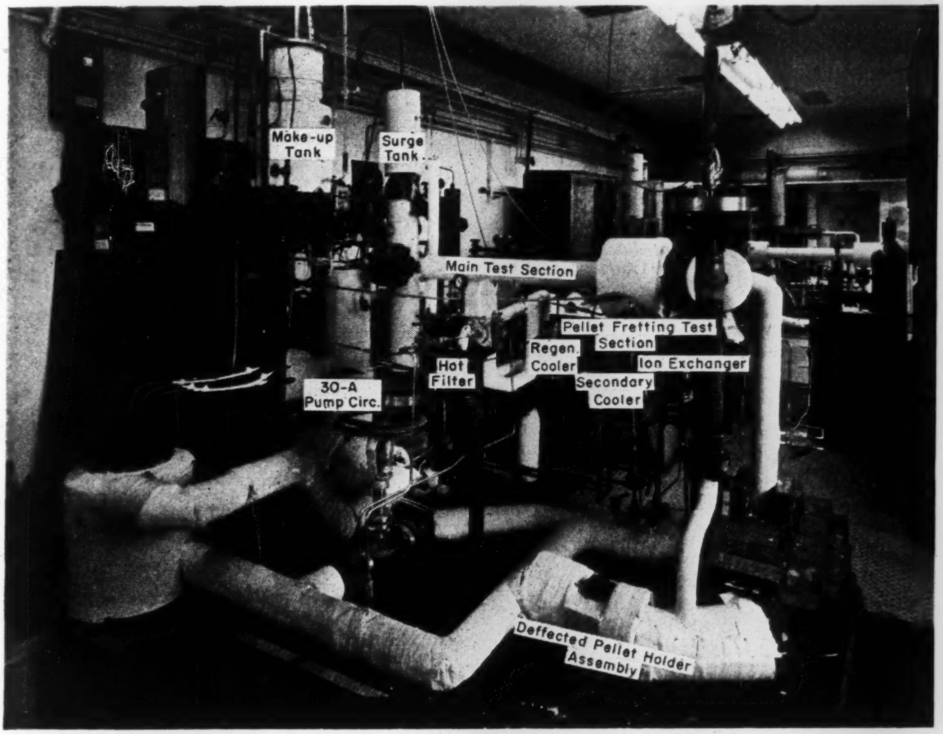

FIG. 2.—Photograph of Typical Bettis Loop Facility.

Details of the cask are shown in Fig. 5. The following procedure was utilized in unloading the can of irradiated pellets from the cask into the loop. A split plug was used for the top shielding plug of the

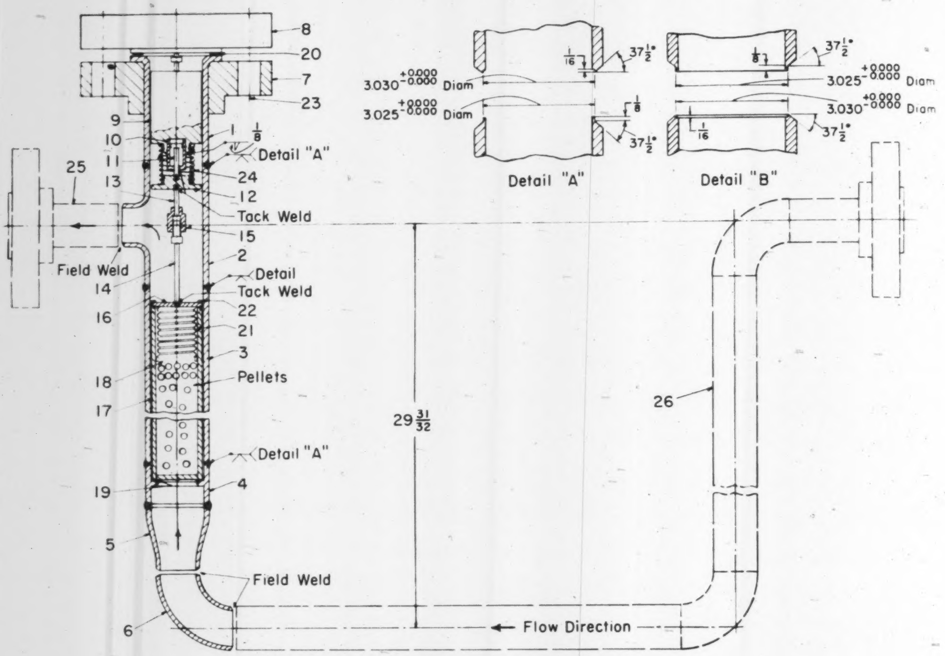
8
20
7
23
9
10
11
1
1/8
Detail "A"
24
12
13
25
Tack Weld
15
Field Weld
2
14
Detail
Tack Weld
16
22
21
3
18
Pellets
17
Detail "A"
19
4
5
Field Weld
6
29 31/32
26
Flow Direction
1/16
37 1/2°
3.030 +0.000 −0.000 Diam
3.025 +0.000 −0.000 Diam
1/8
37 1/2°
Detail "A"
1/8
37 1/2°
3.025 +0.000 −0.000 Diam
3.030 +0.000 −0.000 Diam
1/16
37 1/2°
Detail "B"

FIG. 3.—Test Section—General Assembly.

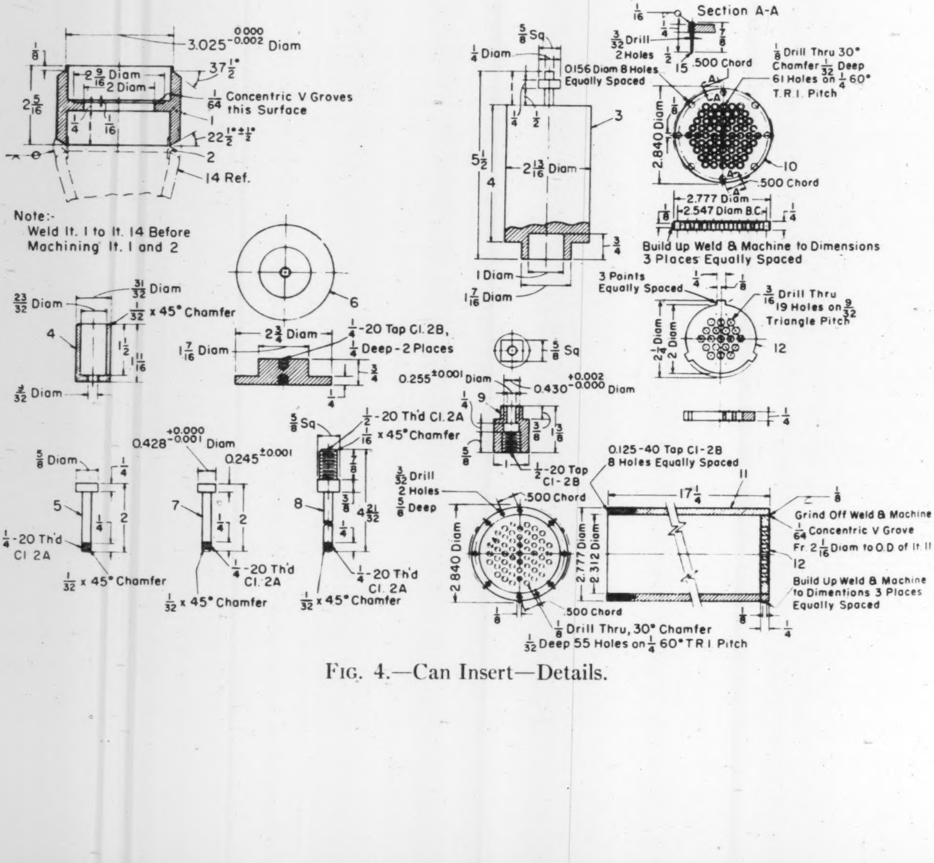
1/8
3.025 0.000 −0.002 Diam
2 9/16 Diam
2 Diam
37 1/2°
1/64 Concentric V Groves this Surface
2 5/16
1
1/4
1/16
22 1/2° ± 1/2°
2
14 Ref.
Note:-
Weld It. 1 to It. 14 Before
Machining It. 1 and 2
5/8 Sq
1/4 Diam
0.156 Diam 8 Holes
Equally Spaced
1/4
1/2
3
5 1/2
4
2 13/16 Diam
3/4
1 Diam
1 7/16 Diam
1/16
Section A-A
3/32 Drill
2 Holes
1/4
7/8
1/2
15
.500 Chord
1/8 Drill Thru 30°
Chamfer 1/32 Deep
61 Holes on 1/4 60°
T.R.I. Pitch
1/8
2.840 Diam
10
.500 Chord
2.777 Diam
2.547 Diam B.C.
1/8
1/4
Build Up Weld & Machine to Dimensions
3 Places Equally Spaced
3 Points
Equally Spaced
1/4
1/8
3/16 Drill Thru
19 Holes on 9/32
Triangle Pitch
2 1/4 Diam
2 Diam
12
1/4
6
23/32 Diam
31/32 Diam
1/32 x 45° Chamfer
4
1 1/2
1 11/16
3/32 Diam
2 3/4 Diam
1/4 -20 Tap Cl.2B,
1/4 Deep-2 Places
1 7/16 Diam
3/4
1/4
5/8 Sq
0.255 ±0.001 Diam
0.430 +0.002 −0.000 Diam
1/4
9
3/8
1 3/8
5/8
1
1/2 -20 Tap
Cl-2B
5/8 Diam
1/4
5
2
1/4
1/4 -20 Th'd
Cl 2A
1/32 x 45° Chamfer
0.428 +0.000 −0.001 Diam
0.245 ±0.001
7
2
1/4
1/4 -20 Th'd
Cl. 2A
1/32 x 45° Chamfer
5/8 Sq
1/2 -20 Th'd Cl.2A
1/16 x 45° Chamfer
8
7/8
3/8
4 21/32
1/4
1/4 -20 Th'd
Cl. 2A
1/32 x 45° Chamfer
3/32 Drill
2 Holes
5/8 Deep
.500 Chord
2.840 Diam
.500 Chord
1/8
1/8 Drill Thru, 30° Chamfer
1/32 Deep 55 Holes on 1/4 60° T R I. Pitch
0.125-40 Tap Cl-2B
8 Holes Equally Spaced
17 1/4
11
1/8
2.777 Diam
2.312 Diam
Grind Off Weld & Machine
1/64 Concentric V Grove
Fr. 2 1/16 Diam to O.D of It. 11
12
Build Up Weld & Machine
to Dimentions 3 Places
Equally Spaced
1/8
1/4

FIG. 4.—Can Insert—Details.

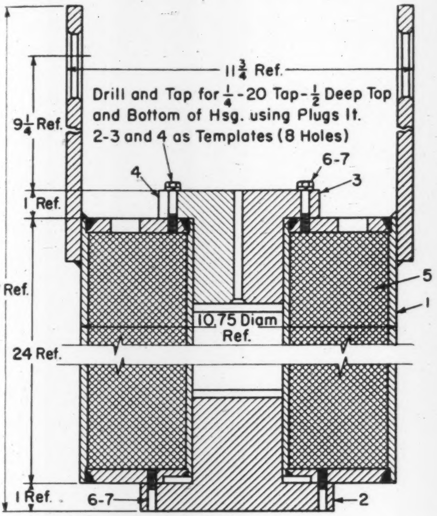
11¾ Ref.
Drill and Tap for ¼-20 Tap-½ Deep Top and Bottom of Hsg. using Plugs It. 2-3 and 4 as Templates (8 Holes)
9¼ Ref.
4
6-7
3
1 Ref.
5
1
37 Ref.
10.75 Diam Ref.
24 Ref.
1 Ref.
6-7
2

FIG. 5.—Lead Cask—General Assembly.

cask so that the can connector rod extending through the cask could be attached to the sliding coupling of the stainless steel shielding plug (see Fig. 3) which was inserted in the loop during loading. A forked lifting tool was slipped over the protrusion on the stainless plug while the cask was in place over the open test section and after removal of the split plug the assembly was lowered in place. The can was removed from the loop by reversing this procedure. This technique minimized exposure to radiation. Much higher levels of activity could have been handled by this procedure with little hazard to personnel.

History and Description of Pellet Bed. A total of 842 Zircaloy-2 pellets consisting of flanged spheres with a polar OD

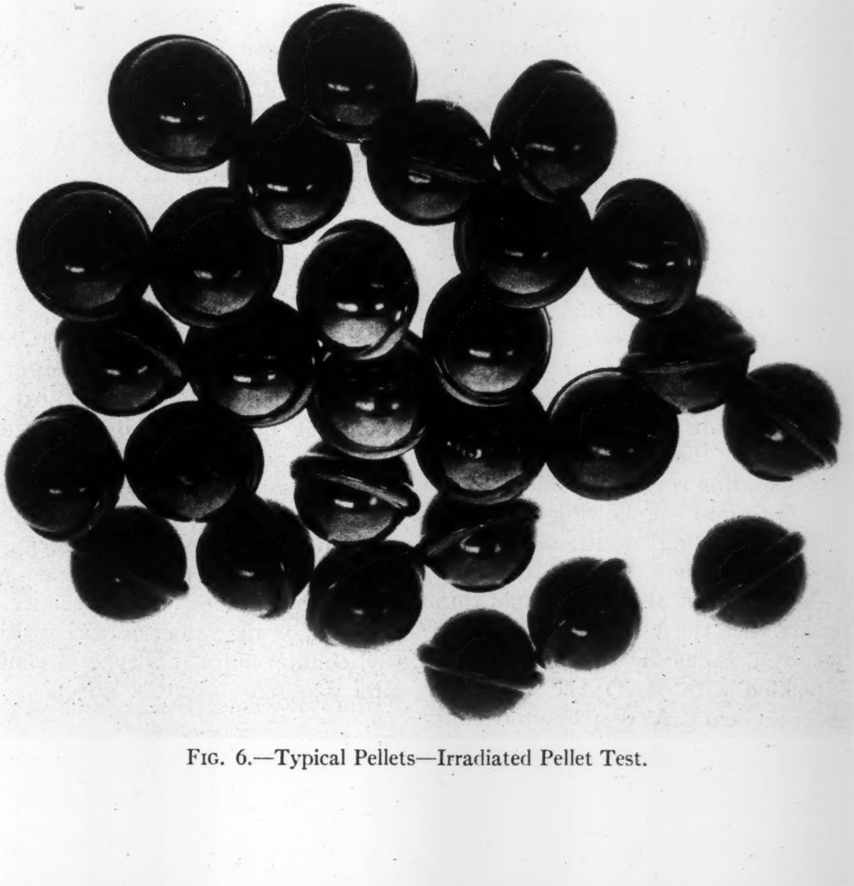

FIG. 6.—Typical Pellets—Irradiated Pellet Test.

of 0.402 in. and an equatorial OD of 0.461 in. was placed in a bed $2\frac{5}{16}$ in. in diameter and 14 in. long. Eighty-five of these pellets, uniformly distributed throughout the bed, were irradiated at the Materials Testing Reactor for one week at a prescribed flux of 1.5×10^{14} neutrons per sq cm per sec. Individual irradiated pellets were placed between layers of nonirradiated pellets within the can, and the bed was vibrated intermittently. The bed was spring loaded by an 85 lb spring placed between the flow distributor plate on top of the bed and the top flow distributor of the can.

All of the pellets had been already tested in the last two of the seven tests referred to previously for a total of 64.5 days in 600 F hydrogenated water as well as three days in 680 F degassed water. Accordingly, the bed as tested presented a preformed corrosion film.

The pellets were fabricated by machining on a turret lathe with form tools. A photograph of typical pellets is shown in Fig. 6.

Test Conditions.—Loop operating conditions during the pellet fretting test were 600 F, 2000 psi, neutral pH, and 25 cc H_2 per kg. An MB-1 resin was in intermittent use in order to maintain a high loop resistivity (nominal 300,000 ohm-cm). A loop flow rate of approximately 18 gal per min (empty column velocity of 1.4 ft per sec) with a corresponding pressure drop of 8.5 psi was utilized during the run. The total test time at operating conditions was 838 hr.

Test Procedure.—Following the test run at the operating conditions referred to above, the pellet bed was removed from the loop and all loop components in contact with the system water during the test run, excluding the 30-A pump, were pickled with HNO_3-HF (15 to 5 weight per cent). A Worthington centrifugal pump was used to circulate the HNO_3-HF through the loop for 1 hr. All pickling solution and flush water used for rinsing the loop were saved for an activity balance.

In addition, the bed was separated into its 85 irradiated and 757 nonirradiated counterparts. Ten representative cold pellets were removed from equidistant points along the length of the bed and pickled with HNO_3-HF in order to obtain estimates of activity deposited on the nonirradiated pellets.

Fifteen pickled pellets with no previous corrosion history served as activity monitors for the 85 irradiated pellets. These 15 pellets were distributed throughout each of five rabbits containing the 85 test pellets during their irradiation at Materials Testing Reactor. One monitor pellet from each rabbit was pickled with HNO_3-HF in order to complete the activity balance.

TABLE I.—ACTIVITY DISTRIBUTION.

Region	Activity, γ Disintegrations per min Corrected to Initial Activity	Per Cent of Total
Loop surfaces	5.03×10^7	62.4
Pellet bed container assembly	0.93	11.5
MB-1 resin	0.30	3.8
Nonirradiated pellets	1.80	22.3
Total	8.06	100

Specific activity of irradiated pellets, disintegration per min per g = 5.40×10^9.

Calculated weight loss due to fretting = 14.9 mg (4.1 mg per sq dm per month)

Liquid samples of the pickling solutions were counted in a gamma-well scintillation counter, and, as a further check, aliquots were evaporated and geiger counted. However, more reliance was placed upon the values obtained with the well counter since this type of counting minimizes self-absorption effects.

RESULTS

Upon completing the balance between

the released activity and the specific activity of the irradiated pellets, the total amount of fretting wear material released from the irradiated pellets was found to be 14.9 mg. Table I is a summary of activity distribution within the test loop and components as determined from the specific activities of the various pickling solutions and their total volumes.

Normalizing the calculated weight loss due to fretting to the surface area of the irradiated pellets, an effective fretting wear rate of 4.1 mg per sq dm per month was found. The significance of this wear rate will be discussed following an analysis of other work performed on beds of Zircaloy pellets.

TABLE II.—SUMMARY OF PELLET FRETTING TESTS I TO VII.

Test	Flow	Test Duration, hr	Samples	Results	ZrO_2 Collected, mg
I.......	up	444	892 pellets	10 per cent showed gray appearance	505
II.......	down	389	692 pellets	10 per cent showed gray appearance	61
III.......	down	141	230 dumbbells	Indistinct local gray markings on 51 per cent	25
IV.......	down	372	230 dumbbells	Indistinct local gray markings 83 per cent	21
			150 pellets	Indistinct local gray markings on 80 per cent	
V.......	down	768	130 pellets 120 dumbbells 130 els	Indistinct local gray markings on 91–92 per cent of samples	75
VI.......	down	780	799 flanged spheres	Local gray markings on 60 per cent of samples	4.3
VII.......	up (spring loaded)	768	842 flanged spheres; same as pretested in Test VI	Percentage of pellets with gray markings not determined	2.9

COMPARISONS WITH PREVIOUS WORK

Most of the comparisons presented herein are based upon an unpublished report by Messrs. D. E. Thomas, S. Kass, and B. G. Schultz, discussions with the authors, and internal summary letters by Messrs. J. Glatter, and S. Kass. Table II is a summary of the pertinent results obtained for the seven pellet fretting tests conducted in Bettis Loop M-1. The environment was essentially the same as that explored for the irradiated pellet fretting test, except for a hydrogen concentration of 100 cu cm per kg as opposed to 25 cu cm per kg in the irradiated pellet test.

The ZrO_2 shown in Table II was that removed from hot filter disks in a by-pass filter across the loop pump. A large proportion of the ZrO_2 released from the bed probably was trapped in system components and could not be accounted for by this method of collection. Since weight changes observed on individual pellets included their weight gain due to normal corrosion, the weight loss due to fretting could only be estimated. The following observations were made:

1. The fact that 10 per cent of the pellets in Tests I and II exhibited large weight losses and gray corrosion products was attributed to possible improper etching and washing prior to test. On the other hand, it was noted that part of the pellet bed could have been fluidized in Test I.

2. Evidence of fretting wear was ap-

parent in the form of dull spots, arcs, and occasionally dull rings on the otherwise lustrous surfaces of the specimens tested in Tests III through VII.

3. The weight gains found for individual pellets were smaller than expected on the basis of tests in static degassed water or in dynamic hydrogenated water. These weight gains could be attributed to fretting wear. For example, from a comparison of actual and expected weight gains in pellet fretting Test V, fretting losses per ball[4] were 0.03 to 0.23 mg whereas for fretting test VII, fretting losses per pellet were found to be 0.03 mg (0.01 g per sq cm) for a portion of the bed previously tested in Test VI and 0.17 mg per pellet for the portion of the bed not tested previously. However, variations in weight changes for some control samples indicated that a more sensitive method of measurement was required.

4. The individual pellets showing fretting wear exhibited one or more fretting sites, in many cases several.

This last observation is important to the analysis of results obtained for the irradiated pellet fretting test. It is interesting to note that in comparison with the Test V and Test VII results given in (3) above, a weight loss of 0.18 mg per pellet was found for an equivalent test period in the irradiated pellet fretting test if the total observed Zr^{95} activity is normalized to the total number (85) of irradiated pellets.

ANALYSIS AND SIGNIFICANCE OF RESULTS—IRRADIATED PELLET TEST

Effect on Clad Integrity.—It is apparent that the consequences of the fretting wear rates found in this test are related to the number of fretting sites involved. Table III below relates the observed material release as determined through the activity balance (14.9 mg) to penetration within the pellet, assuming the fretted volume to consist of a spherical segment. The worst possible case would consist of only two pellets within the entire bed fretting at one site.

The penetration per site in Table III is expressed by the relationship:

$$V = \frac{1}{3}\pi h^2(3R - h) \ldots\ldots\ldots (1)$$

where:

V = volume of fretted material,
h = depth of penetration, and
R = radius of sphere.

From the results presented above, it is safe to assume that at least 50 per cent of the irradiated pellets contacted one or more fretting sites per pellet. A maximum of twelve contact points per pellet could be obtained in a hexagonal close packed bed. Accordingly, the worst possible case, (a)—Table III, is not considered to be realistic; case (c) is probably more representative of the real condition. A visual examination of individual pellets did not indicate any localized attack corresponding to a 10.6 mil penetration.

TABLE III.—CLADDING PENETRATION PER FRETTING SITE.

Number of Irradiated Pellets Fretting	Irradiated Pellets, per cent	Number of Fretting Sites per Pellet	840 hr Penetration per Site, mils = h
(a) 2........	2.4	1	10.6
(b) 20........	24	1	3.3
(c) 42........	50	2	1.6
(d) 85........	100	1	1.6
(e) 85........	100	2	1.1

Extrapolation of the amount of fretting wear to operation for longer periods must be evaluated in terms of fretting wear theories[5] and the geometry involved. According to an analysis by Uhlig, *et al*, the total wear of fretting surfaces is attributable to a combination of corrosion and mechanical abrasion. For Uhlig's model, movement over the contact points has a wiping action on the corrosion film resulting in the exposure of clean surfaces. The exposed fresh surfaces corrode in turn and new scale is subsequently removed by wiping at the contact points. The use of this model by Uhlig, *et al*, resulted in a relationship showing total wear to be a parabolic function of the applied constant area load and a linear function of time. The contribution due to corrosion was overshadowed by mechanical wear at high frequencies, and the total wear for this high frequency case was predicted to be a linear function of the load between constant area fretting surfaces.

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[4] The els and dumbells were made up of connected balls similar to the individually tested spheres.

[5] H. H. Uhlig *et al.*, "A Fundamental Investigation of Fretting Corrosion," NACA TM 3029, Dec., 1953.

For a spherical pellet, it is apparent that the area of contact will increase with depth of penetration. Assuming a constant force between the fretting surfaces, the total amount of wear would be linear with time, if the wear rate is linear with load on the contact surfaces as predicted by the Uhlig analysis with a low corrosion contribution. In support of the hypothesis it can be seen that the contact pressure is inversely proportional to the square of the contact area radius while the material fretted is proportional to the square of the contact area radius.

Accordingly, if the wear rate is parabolic with the contact surface load, then a decrease in fretting rates with time would be expected. Work conducted at the Bettis site with small loads on Zircaloy fretting surfaces has provided further support for this parabolic relationship. Evidently the assumption that fretting wear for spherical pellets is linear with

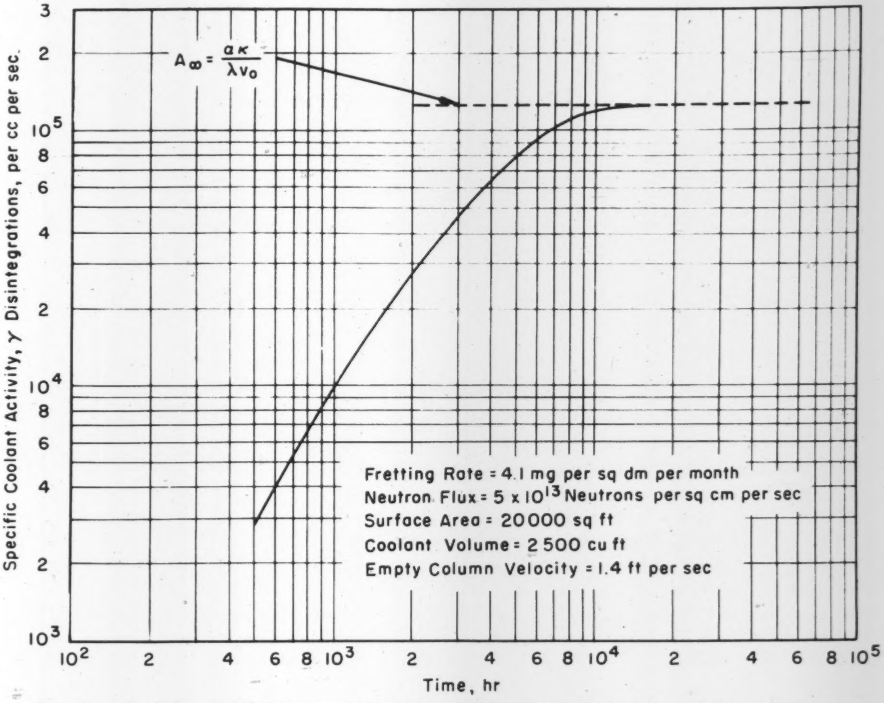

FIG. 7.—Activity Build-Up due to Fretting of Zircaloy-2 Pellets in a Reference Core.

time is pessimistic, even if high frequency mechanical abrasion predominates in the bed. The significance of this analysis with respect to penetration depth within the cladding is shown below.

Since the total fretting wear according to the above analysis can be assumed to be, at worst, linear with time, rates of penetration depth may be determined. Given Eq 1, it can be easily shown that for small values of h,

$$\frac{dh}{dt} = \frac{k}{2\pi hR - \pi h^2} \approx \frac{k}{2\pi hR} \quad \ldots\ldots (2)$$

where:

k = constant wear rate, and
t = time

From Eq 2 it is apparent that the rate of penetration is inversely proportional to the penetration distance for significant values of h. By integrating Eq 2, it can be shown that

$$h \alpha t^{1/2} \quad \ldots\ldots\ldots\ldots\ldots (3)$$

Accordingly, for a total operating time ten times that shown in cases (c or d) in Table III, approximately 1 yr, a depth of penetration of $(1.6)(10)^{1/2}$, or 5 mils, would be expected.

Pending further tests to establish the number of sites involved, we conclude that the effect of fretting corrosion on clad integrity in a bed of Zircaloy pellets is not serious.

Effect of Activity Release on Acessibility to Loop Compartments.—A plot of activity build-up as a function of time with a reference core is shown in Fig. 7 for a fretting rate of 4.1 mg per sq dm per month and no purification or re-irradiation of released material. The calculations for specific coolant activities were based upon the assumption that all of the Zr^{95} activity released into the loop is suspended in the system coolant. This, of course, is not a real case, but the end result in terms of accessibility is essentially the same even though a large portion of the activity is deposited on the pipe walls. The assumption was used only to establish a reference for discussion. The following bases were utilized in the calculations.

Fretting rate............	4.1 mg per sq dm per month
Total surface area of pellets..................	20 000 sq ft
Primary coolant volume..	2500 cu ft
Neutron flux............	5×10^{13} neutrons per sq cm per sec

The curve plotted in Fig. 4 was based on the equations:

$$\frac{d(N_i)_m}{dt} = N\sigma_a\varphi - \lambda(N_i)_m \quad \ldots\ldots (4)$$

$$\frac{d(N_i)}{dt} = \frac{\alpha(N_i)_m}{V_o} - \lambda(N_i) \quad \ldots\ldots (5)$$

and the solution to Eqs. 4 and 5:

$$N_i = \frac{\alpha K}{\lambda V_o} e^{-\lambda t} \left[\frac{1}{\lambda} e^{\lambda t} - \frac{1}{\lambda} - t\right] \ldots (6)$$

$$\lim_{t \to \infty} (N_i) = \frac{\alpha K}{\lambda^2 V_o} = A/\lambda \quad \ldots\ldots (7)$$

where:

$(N_i)_m$ = concentration of Zr^{95} in parent metal, atoms Zr^{95} per cu cm metal,
(N_i) = concentration of Zr^{95} in loop water, atoms Zr^{95} per cu cm coolant,
N = atoms Zr^{94} per cu cm metal,
t = time of operation with the given neutron flux, sec,
σ_a = absorption cross section of Zr^{94}, sq cm,
φ = neutron flux, neutrons per cm per sec,
λ = decay constant, per sec,
α = rate of parent metal release to coolant, cu cm per sec,
V_o = primary coolant volume, cu cm,
K = $N\sigma_a\varphi$, atoms Zr^{95} per sec per cu cm metal, and
A = $(N_i)\lambda$, γ disintegrations per sec per cu cm coolant.

As can be seen in Fig. 7, a saturation level of 1.27 × 10^5 γ disintegrations per sec per cu cm coolant is reached with a release rate of 4.1 mg per sq dm per month. Less than one-half this value is reached after 3000 hr of operation. Tolerable levels on the order of 5 × 10^4 γ disintegration per sec per cu cm have been proposed for accessibility to a loop compartment with 2.59 hr Mn^{56} as a major contributor with a γ energy of 2.06 Mev compared to a softer γ of 0.73 Mev for Zr^{95}. Shielding calculations based on available relationships indicate that with the total Zr^{95} coolant activity corresponding to a saturation level of 1.27 × 10^5 γ disintegrations per sec per cu cm of coolant, when uniformly distributed over the surfaces of a large reactor primary system, levels less than 50 mr per hr would be expected at a distance of 1 ft from 18 in. primary system piping. Accordingly, the levels shown in Fig. 5 are not serious with respect to accessibility requirements.

Re-irradiation of the released ZrO_2 presupposes transportability of the fretted material in the coolant stream. If this transportability occurs,[6] the contribution due to re-irradiation of the released ZrO_2 in the reactor core would be considerably less than that due to the removal of suspended activity in the by-pass purification system. In fact, transportability would result in lower activities with continued purification and re-irradiations.

The maximum specific Zr^{95} activity that the coolant would build up with re-irradiation and no purification corresponds to the saturation activity shown in Fig. 7. The effect of continuous purification on a system in which activity is transported is shown by the relationship:

$$\frac{d(N_i)}{dt} = \frac{\alpha(N_i)_m}{V_o} - \lambda(N_i) - E\beta(N_i) \ldots (8)$$

where:

β = purification removal constant = $\dfrac{\text{purification flow rate}}{\text{total primary coolant volume}}$, and

E = efficiency of purification system ≈ 1 for D.F.'s greater than 5 with MB-1 resin.

and the remaining items defined as for Eqs 4 through 7.

The solution to Eq 8 is:

$$(N_i) = \frac{\alpha K}{\lambda V_o}\left[\frac{1}{(\lambda + E\beta)} - \frac{1}{E\beta}e^{-\lambda t} + \frac{\lambda e^{-(\lambda+E\beta)t}}{E\beta(\lambda + E\beta)}\right] \ldots (9)$$

and at t = ∞

$$(N_i) = \frac{\alpha K}{\lambda V_o}\left[\frac{1}{\lambda + E\beta}\right] = A/\lambda \ldots (10)$$

Substituting the appropriate values in (10), a saturation level of only 1.5 × 10^2 γ disintegrations per cu cm per sec is reached. Of course, this calculation is not based on the assumption that such low levels can actually be achieved through purification of the main coolant stream; however, it does illustrate that lower levels than those shown in Fig. 7 can be expected, depending on the percentage of released material suspended in the coolant, and that re-irradiation of released material is negligible compared to the effect of purification. For 1 per cent of the specific activity reaching the purification unit by-pass at infinite time with continuous purification, the coolant specific activity would be 1.3 × 10^4 γ disintegrations per cu cm per sec.

SUMMARY AND CONCLUSIONS

The results of this test showed a fretting rate of 4.1 mg per sq dm per month for a packed bed of Zircaloy pellets. Pending further investigations to establish the number of fretting sites per pellet (or per unit bed) involved in the fretting phenomenon, it is concluded that this rate is not serious with respect to clad integrity. Activity levels reached in the primary system with the given fretting rate are not serious with respect to accessibility to the loop compartment.

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[6] Work conducted at the Bettis Site indicates that a large proportion of the total material release will be trapped in the system.

Acknowledgments:

The work reported herein was performed for the Atomic Energy Commission under Contract AT-11-1-GEN-14. The authors wish to express their appreciation to Dr. D. E. Thomas and Messrs. J. Glatter and S. Kass for many valuable contributions.

INTERGRANULAR CORROSION RESISTANCE OF LOW-CARBON AUSTENITIC CHROMIUM-MANGANESE-NICKEL STEELS*

By W. O. Binder,[1] J. Thompson,[2] and C. R. Bishop[3]

Synopsis

The austenitic chromium-manganese-nickel steels become susceptible to intergranular corrosion when heated in the temperature range of 425 to 815 C. The immunization of these steels with columbium or titanium introduces metallurgical complications, and in view of this, the possibility of gaining control of intergranular corrosion by lowering carbon was investigated. This approach eliminates the problems associated with steels containing columbium and titanium, since the low-carbon steels can be made fully austenitic by increasing nitrogen.

The study included steels containing 18 per cent chromium, 7 to 11 per cent manganese, 4 to 10 per cent nickel, 0.4 per cent silicon, 0.015 to 0.045 per cent carbon, and 0.22 per cent nitrogen. The manganese content of the steels was, with one exception, greater than the nickel content. The permissible carbon limits for resistance to intergranular corrosion, after heating at 650 C, were found to decrease as the sum of the manganese and nickel contents of the steel increased above 14 per cent. The steels containing up to about 0.05 per cent carbon and 14 per cent manganese plus nickel were found resistant to intergranular attack after 1 hr at 650 C, and those containing 0.02 to 0.025 per cent carbon and not more than 14 per cent manganese plus nickel were resistant after 24 hr at 650 C. Increasing the manganese-plus-nickel content to 18 per cent required decreasing these limits to about 0.035 and 0.02 per cent carbon, respectively.

The austenitic chromium-manganese-nickel steels become susceptible to intergranular corrosion when heated in the temperature range of 425 to 815 C (**1**).[4] The immunization of these steels with columbium or titanium introduces metallurgical complications. The formation of delta ferrite, due to the removal of carbon and nitrogen from solution by columbium or titanium, may lead to hot-working difficulties, and the subsequent transformation of this phase to sigma phase when the steel is exposed to elevated temperatures may produce brittleness and reduced corrosion resistance. In low-nickel steels, the austenite is subject to transformation to low-temperature ferrite when titanium is present, and the

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[4] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 920.

TABLE I.—COMPOSITION OF STEELS INVESTIGATED AND RESULTS OF BOILING 65 PER CENT NITRIC ACID TESTS ON SPECIMENS HEATED AT 550 C.

Heat	Composition per cent						Third Period Corrosion Rate, penetration, in. per month	
	Chromium	Manganese	Nickel	Silicon	Carbon[a]	Nitrogen[a]	Heated ½ hr at 1075 C and air-cooled[b]	Heated ½ hr at 1075 C and Air-Cooled. Reheated 100 hr at 550 C[b]
S318	18.25	7.0	4.5	0.4	0.017	0.22	N.T.	N.T.
S319	18.25	7.0	4.5	0.4	0.020	0.22	N.T.	N.T.
S286	18.25	7.0	4.5	0.4	0.028	0.22	N.T.	N.T.
S287	18.25	7.0	4.5	0.4	0.030	0.21	N.T.	N.T.
S288	18.25	7.0	4.5	0.4	0.036	0.21	N.T.	N.T.
S289	18.25	7.0	4.5	0.4	0.040	0.22	N.T.	N.T.
S324	18.25	7.0	5.0	0.4	0.015	0.22	N.T.	N.T.
S325	18.25	7.0	5.0	0.4	0.024	0.22	0.00099	0.0020
S326	18.25	7.0	5.0	0.4	0.028	0.22	0.0010	0.0011
S327	18.25	7.0	5.0	0.4	0.029	0.22	0.00099	0.0011
S328	18.25	7.0	5.0	0.4	0.034	0.22	0.0010	0.0092
S329	18.25	7.0	5.0	0.4	0.043	0.22	0.0010	0.073
S151	18.25	7.0	6.0	0.4	0.010	0.22	0.0008	0.00085
S152	18.25	7.0	6.0	0.4	0.021	0.23	0.00082	0.00088
S153	18.25	7.0	6.0	0.4	0.026	0.22	0.00082	0.00093
S154	18.25	7.0	6.0	0.4	0.028	0.22	0.00089	0.0016
S198	18.25	7.0	6.0	0.4	0.037	0.22	0.00094	0.052
S155	18.25	9.0	4.0	0.4	0.013	0.20	0.0010	0.0011
S157	18.25	9.0	4.0	0.4	0.025	0.23	0.0011	0.0014
S158	18.25	9.0	4.0	0.4	0.027	0.22	0.0010	0.0014
S156	18.25	9.0	4.0	0.4	0.029	0.22	0.0012	0.0027
S311	18.25	9.0	4.0	0.4	0.037	0.22	0.0013	0.0027
S199	18.25	9.0	4.0	0.4	0.043	0.22	0.0013	0.134
S320	18.25	9.0	4.5	0.4	0.012	0.22	0.0011	0.0010
S321	18.25	9.0	4.5	0.4	0.023	0.22	0.0010	0.0010
S293	18.25	9.0	4.5	0.4	0.028	0.22	0.0013	0.0052
S292	18.25	9.0	4.5	0.4	0.034	0.21	0.0011	0.0070
S294	18.25	9.0	4.5	0.4	0.040	0.22	0.0011	0.014
S159	18.25	9.0	5.0	0.4	0.010	0.22	0.0010	0.0010
S160	18.25	9.0	5.0	0.4	0.020	0.22	0.00093	0.0021
S161	18.25	9.0	5.0	0.4	0.021	0.21	0.00095	0.0014
S260	18.25	9.0	5.0	0.4	0.026	0.21	0.00099	0.011
S200	18.25	9.0	5.0	0.4	0.035	0.23	0.00094	0.0048
S162	18.25	9.0	5.0	0.4	0.036	0.23	0.0011	0.083
S163	18.25	9.0	6.0	0.4	0.010	0.21	0.00078	0.00081
S164	18.25	9.0	6.0	0.4	0.021	0.22	0.00086	0.00091
S165	18.25	9.0	6.0	0.4	0.024	0.22	0.00095	0.00096
S166	18.25	9.0	6.0	0.4	0.027	0.22	0.0010	0.0014
S201	18.25	9.0	6.0	0.4	0.036	0.23	0.0011	0.011
S167	18.25	9.0	7.0	0.4	0.015	0.22	0.00078	0.00070
S313	18.25	9.0	7.0	0.4	0.020	0.22	0.00073	0.00070
S169	18.25	9.0	7.0	0.4	0.028	0.23	0.00083	0.0020
S170	18.25	9.0	7.0	0.4	0.034	0.22	0.00087	0.0093
S202	18.25	9.0	7.0	0.4	0.040	0.22	0.00081	0.131

TABLE I.—*Concluded.*

Heat	Composition, per cent						Third Period Corrosion Rate, penetration, in. per month	
	Chromium	Manganese	Nickel	Silicon	Carbon[a]	Nitrogen[a]	Heated ½ hr at 1075 C and air-cooled	Heated ½ hr at 1075 C and Air-Cooled. Reheated 100 hr at 550 C
S171.......	18.25	9.0	10.0	0.4	0.015	0.21	0.00053	0.0006
S262.......	18.25	9.0	10.0	0.4	0.018	0.19	0.00054	0.010
S173.......	18.25	9.0	10.0	0.4	0.025	0.22	0.00062	0.013
S172.......	18.25	9.0	10.0	0.4	0.027	0.22	0.00057	0.007
S174.......	18.25	9.0	10.0	0.4	0.033	0.23	0.00064	0.017
S203.......	18.25	9.0	10.0	0.4	0.042	0.22	0.00069	0.11
S322.......	18.25	11.0	4.5	0.4	0.019	0.22	0.0011	0.0010
S323.......	18.25	11.0	4.5	0.4	0.022	0.22	0.0011	0.0011
S298.......	18.25	11.0	4.5	0.4	0.026	0.22	0.0011	0.0012
S299.......	18.25	11.0	4.5	0.4	0.034	0.22	0.0010	0.020
S300.......	18.25	11.0	4.5	0.4	0.039	0.22	0.0011	0.067
S301.......	18.25	11.0	4.5	0.4	0.04	0.21	0.0012	0.087
S179.......	18.25	11.0	5.0	0.4	0.014	0.21	0.00094	0.00095
S180.......	18.25	11.0	5.0	0.4	0.022	0.23	0.0011	0.0010
S181.......	18.25	11.0	5.0	0.4	0.025	0.22	0.0010	0.017
S182.......	18.25	11.0	5.0	0.4	0.033	0.23	0.0011	0.046
S205.......	18.25	11.0	5.0	0.4	0.035	0.23	0.0011	0.039
S183.......	18.25	11.0	6.0	0.4	0.015	0.21	0.00079	0.0008
S184.......	18.25	11.0	6.0	0.4	0.023	0.24	0.00086	0.0011
S185.......	18.25	11.0	6.0	0.4	0.027	0.23	0.00088	0.044
S186.......	18.25	11.0	6.0	0.4	0.035	0.22	0.00090	0.033
S206.......	18.25	11.0	6.0	0.4	0.04	0.23	0.00091	
S187.......	18.25	11.0	7.0	0.4	0.016	0.21	0.00066	
S265.......	18.25	11.0	7.0	0.4	0.021	0.20	0.00068	0.00079
S189.......	18.25	11.0	7.0	0.4	0.026	0.22	0.00081	0.018
S188.......	18.25	11.0	7.0	0.4	0.029	0.23	0.00078	0.027
S190.......	18.25	11.0	7.0	0.4	0.038	0.23	0.00084	0.097

[a] Actual content.
[b] N.T. = Not tested for intergranular corrosion resistance because of ingot porosity.

steel may lose its non-magnetic characteristics.

In view of these disadvantages, the possibility of gaining control over intergranular corrosion by lowering carbon was investigated. This approach eliminates the problem of austenite instability since the low-carbon steels can be maintained fully austenitic by the simple expedient of increasing the nitrogen. Nitrogen can be utilized for this purpose because it does not promote intergranular corrosion as does carbon (2). This method has been successfully applied to the austenitic chromium-nickel steels. The carbon content of the chromium-nickel steels must be decreased below 0.02 per cent for complete immunity; however, for many applications, reducing carbon to about 0.03 per cent provides sufficient immunity to permit the steels to be welded without subsequent solution heat treatment.

The development of steels having maximum tolerance for carbon is desirable from a manufacturing standpoint. The steels investigated contained 18 per cent chromium, 7 to 11 per cent manganese, 4 to 10 per cent nickel, 0.4 per cent silicon, 0.015 to 0.045 per cent carbon, and 0.22 per cent nitrogen. The nickel and manganese contents were varied rather widely to evaluate the influence of these elements on the limits of carbon permissible while maintaining immunity to intergranular corrosion. With one exception, the steels contained more manganese than nickel.

The steels were tested in boiling 65 per cent nitric acid, boiling acidified copper sulfate solution, and in 10 per cent nitric acid—3 per cent hydrofluoric acid solution at 70 C after heat treatment at temperatures between 300 and 650 C to induce intergranular corrosion susceptibility. These test media and the heat treatments at 550 and 650 C have been found useful for evaluating the intergranular corrosion resistance of 0.03 per cent carbon austenitic chromium-nickel steels (2, 3).

The tolerance for carbon after heating at 650 C was found to decrease as the sum of the manganese-plus-nickel contents of the steel increased. Steels containing up to about 0.05 per cent carbon and 14 per cent manganese plus nickel are resistant to intergranular attack after 1 hr at 650 C, and those containing 0.02 to 0.025 per cent carbon and not more than 14 per cent manganese plus nickel are resistant after 24 hr at 650 C. Increasing the manganese-plus-nickel content to 18 per cent, decreases these limits to 0.035 and 0.02 per cent carbon, maximum for 1 hr and 24 hr, respectively.

Since current arc-melting practice limits carbon to about 0.025 per cent minimum, the requirement of complete immunity imposes a limit of about 14 per cent on the manganese-plus-nickel content of the steel. Although the requirements for partial immunity are less restrictive, from a manufacturing standpoint, the gain in carbon tolerance associated with a decrease in alloy content also warrants restricting the manganese-plus-nickel content of these steels to about 14 per cent.

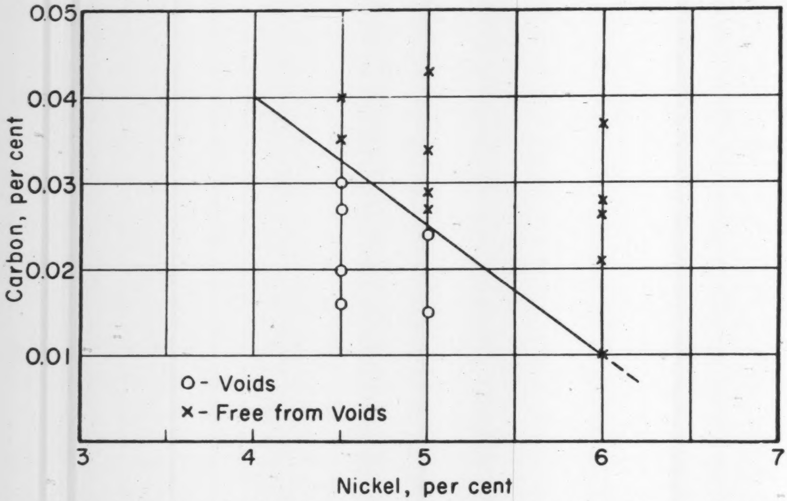


FIG. 1.—Effect of Carbon and Nickel on the Soundness of 18 per cent Chromium, 7 per cent Manganese, 0.22 per cent Nitrogen Steels.

PREPARATION OF STEELS

The chemical composition of the austenitic chromium-manganese-nickel steels requires careful balance to avoid the formation of delta ferrite (**4**), and the steels normally contain about 0.15 per cent nitrogen to promote the formation of austenite. The nitrogen content of the steels studied in this investigation was arbitrarily set at 0.22 per cent to avoid porosity and to help maintain an austenitic structure as carbon was decreased.

The steels were made by induction melting in a magnesia-lined furnace. Each charge weighed 15 lb and consisted of Armco ingot iron, low-carbon ferrochromium, low-carbon 2 per cent nitrogen-ferrochromium, 6 per cent nitrogen-electrolytic-manganese metal, electrolytic nickel, low-carbon manganese metal, and silicon metal. Table I gives the nominal composition and actual carbon and nitrogen contents of the steels.

The ingots were hot-rolled, without prior forging, at an initial temperature of 1150 to 1175 C into $\frac{1}{4}$-in. plate and $\frac{1}{16}$-in. thick strip.

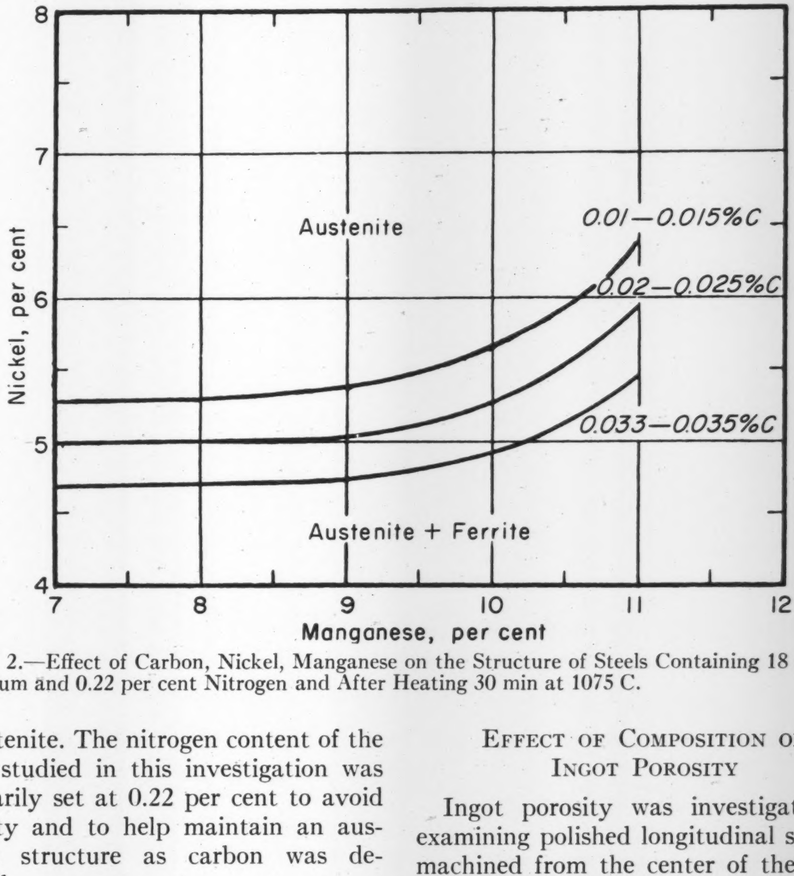


FIG. 2.—Effect of Carbon, Nickel, Manganese on the Structure of Steels Containing 18 per cent Chromium and 0.22 per cent Nitrogen and After Heating 30 min at 1075 C.

EFFECT OF COMPOSITION ON INGOT POROSITY

Ingot porosity was investigated by examining polished longitudinal sections machined from the center of the upper one-third section of the ingot.

No evidence of porosity was found except in some of the 7 per cent manganese steels. The porosity is presumably due to the fact that the solid solubility of nitrogen was exceeded in these steels. As shown in Fig. 1, the porosity was critically related to the carbon and nickel contents of the steel.

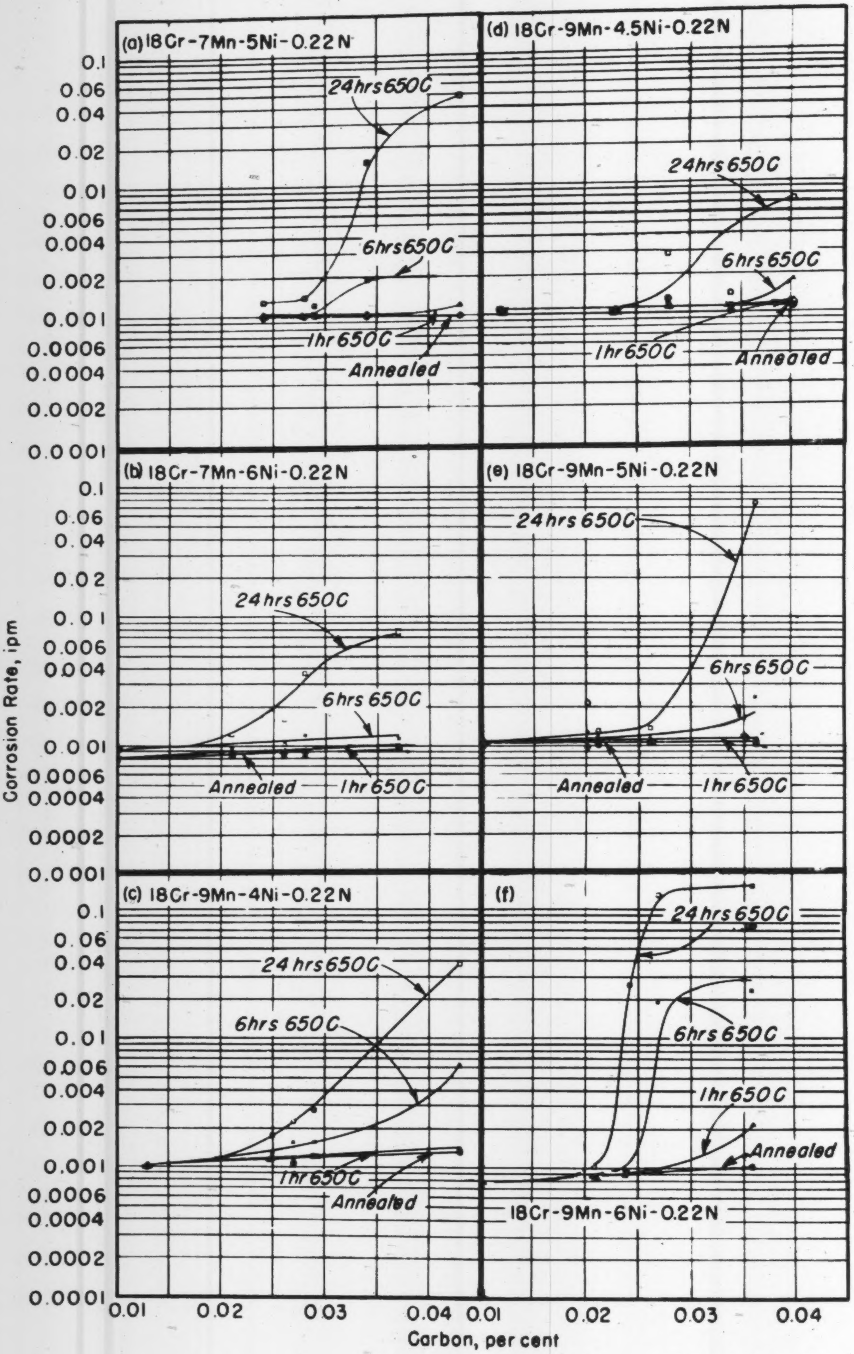
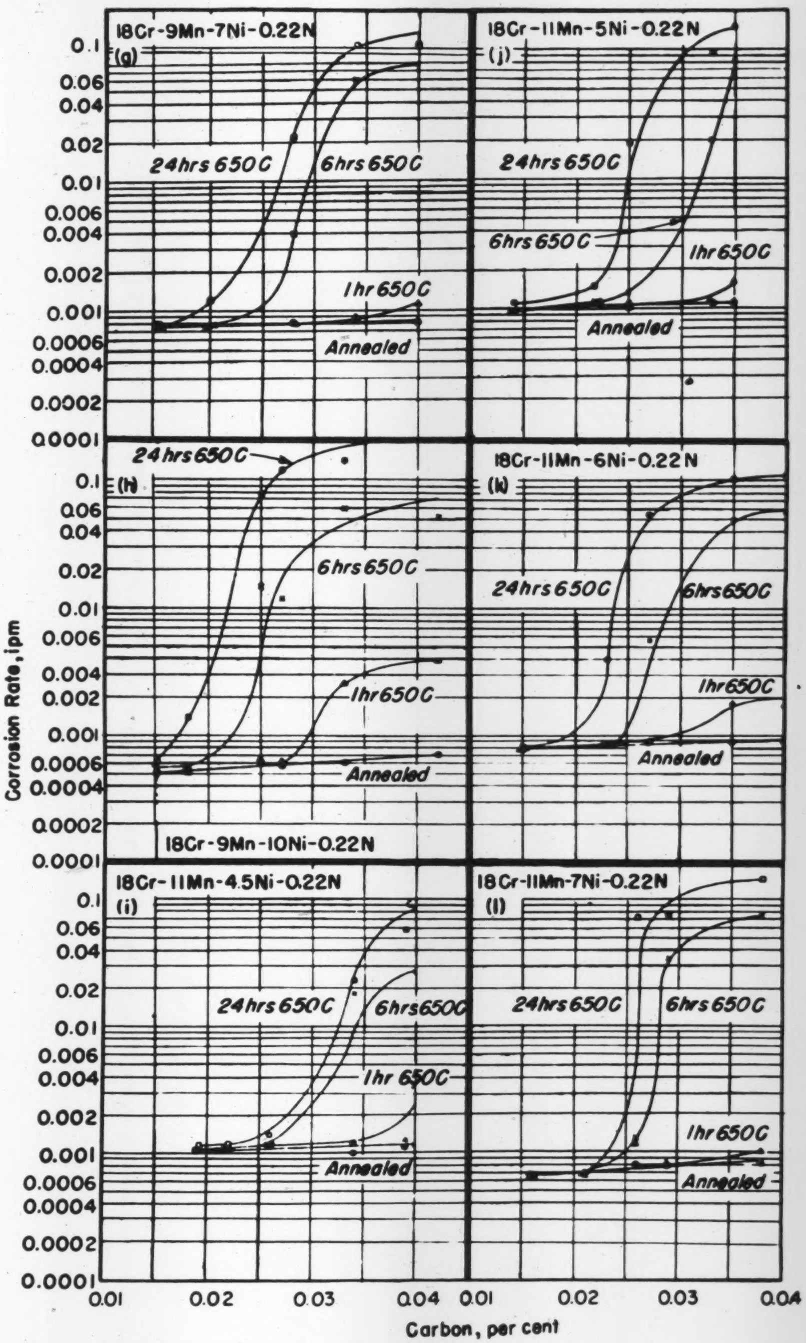


FIG. 3.—Corrosion Resistance of Steels in Boiling



65 per cent Nitric Acid After Heating at 650 C.

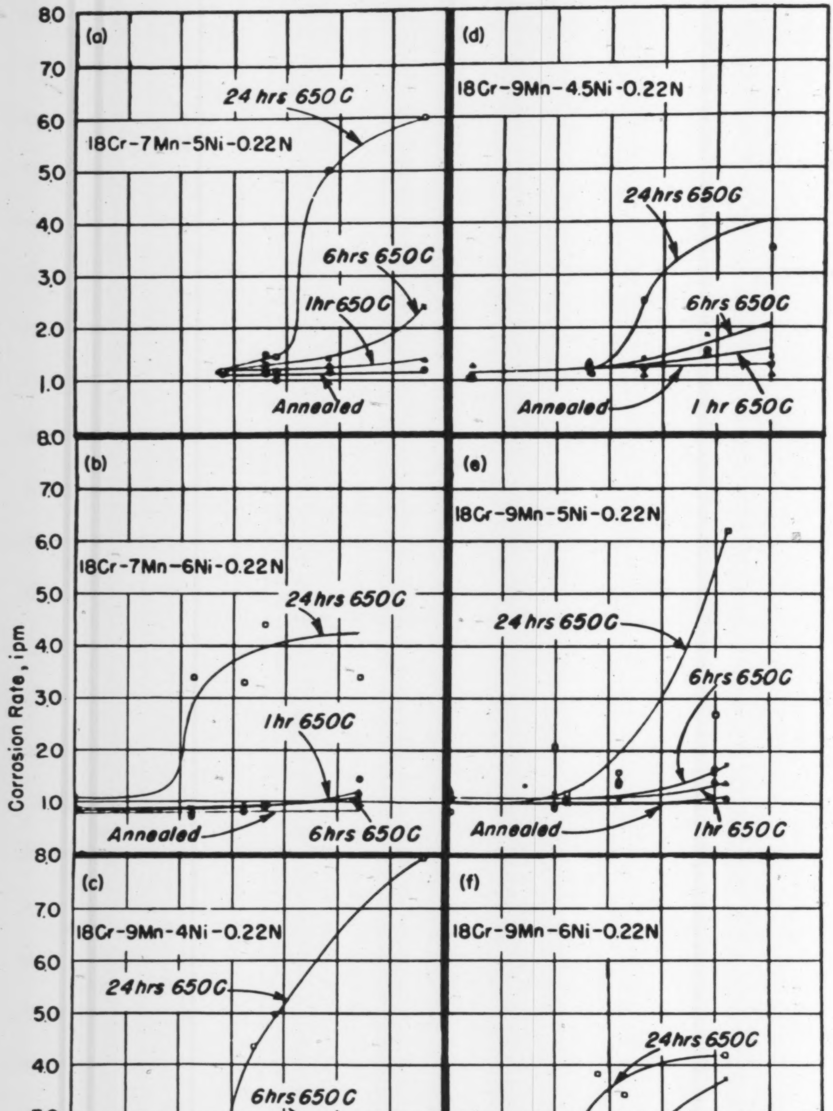
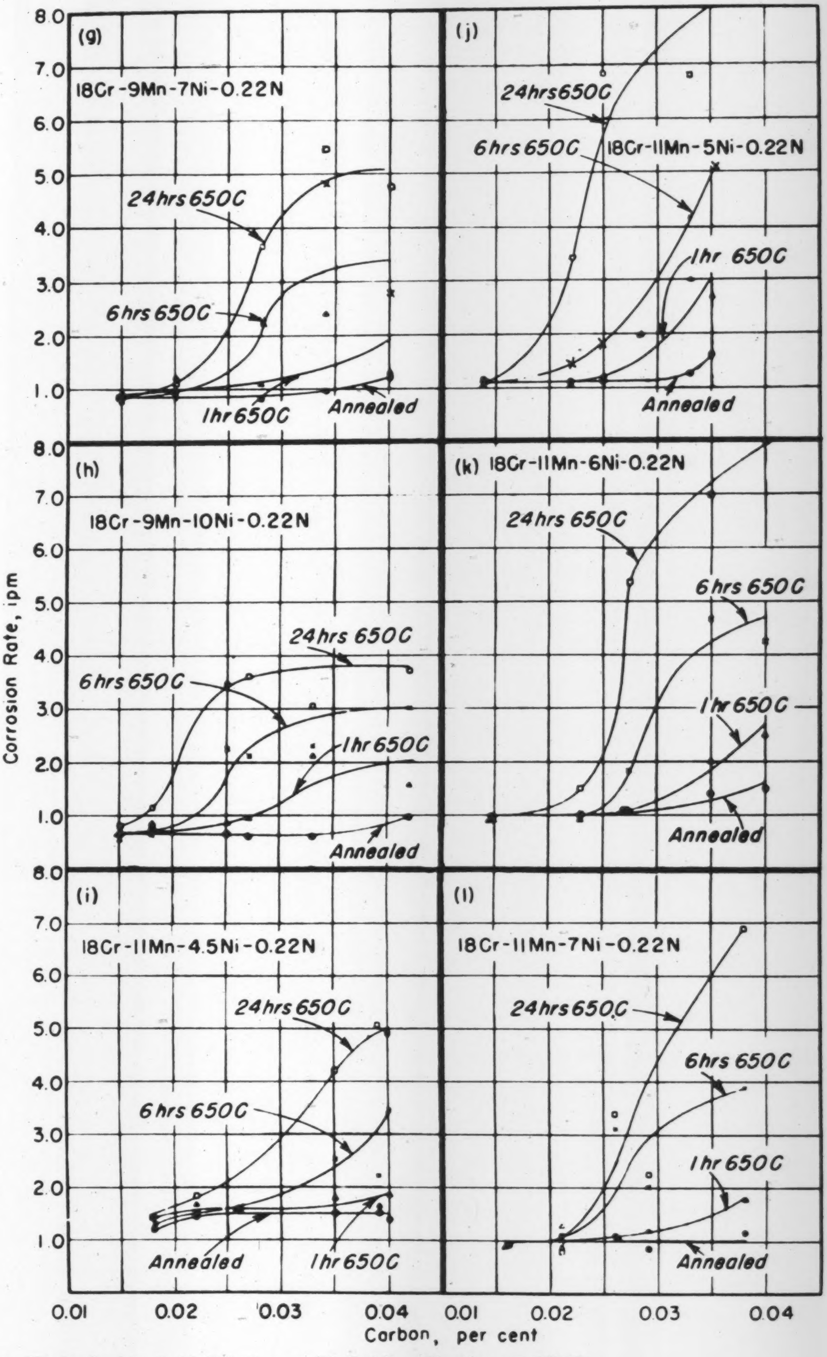


FIG. 4.—Corrosion Resistance of Steels in 10 per cent Nitric Plus 3



per cent Hydrofluoric Acid Solution at 70 C After Heating at 650 C.

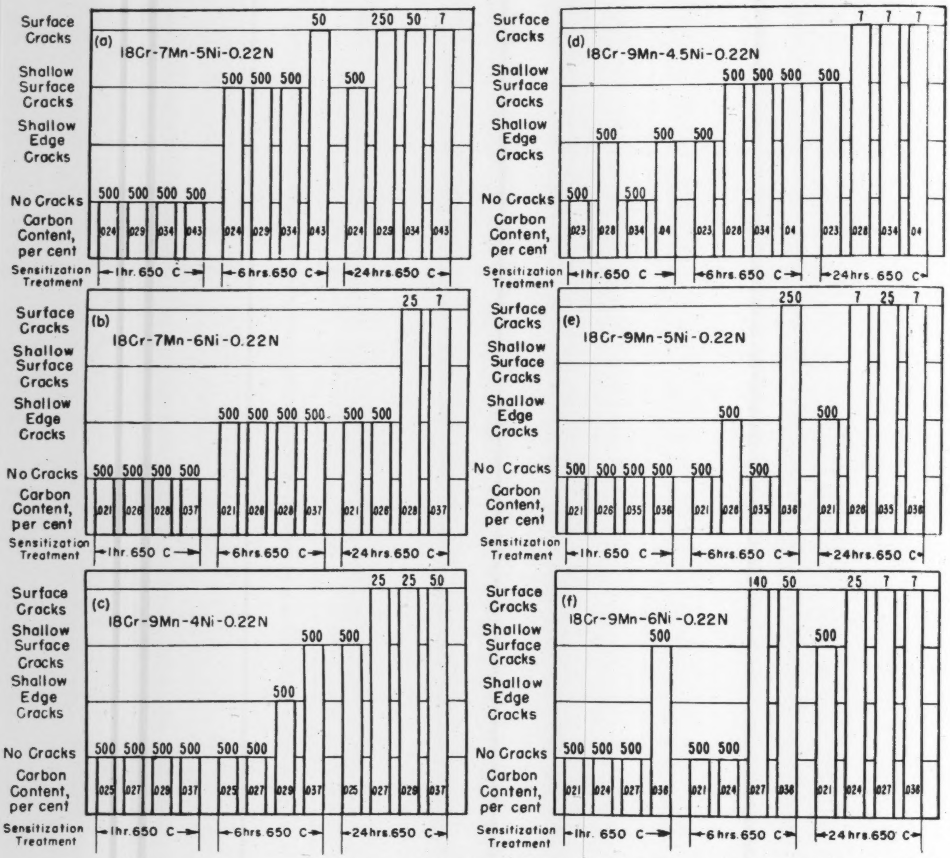


FIG. 5.—Resistance of Steels to Intergranular Corrosion in Boil-
The immersion times are shown

EFFECT OF COMPOSITION ON DELTA FERRITE CONTENT

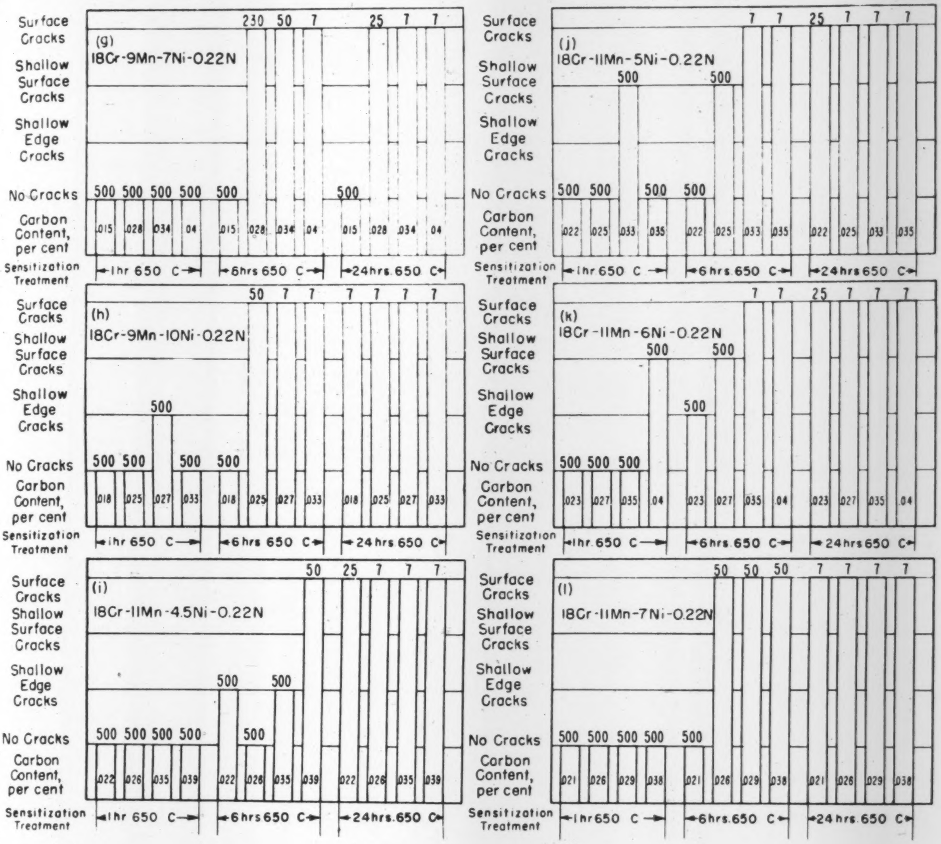
The ferrite content of the steels was estimated by a magnetic pull test previously described (4), and by metallographic examination. The information gained from these tests was used to construct the structural diagram shown in Fig. 2.

The amount of ferrite increased with decreasing carbon and nickel. It also increased with the addition of more than 9 per cent manganese. The ferrite-forming behavior of manganese is in agreement with earlier studies which showed that, when manganese is increased above about 4 per cent, it acts as an austenite stabilizer rather than an austenite former, and when increased above about 9 per cent, it acts as a ferrite former (4). The reason for this dual behavior of manganese has not been established.

TEST CONDITIONS

The intergranular corrosion tests were conducted on specimens heated 30 min at 1075 C and air-cooled, then reheated 1, 6, or 24 hr at 650 C; 100 hr at 550 C; 8 weeks at 400 C; or 16 weeks at 300 C.

The corrosion test solutions consisted of boiling 65 per cent nitric acid, 10 per



ing Acidified Copper Sulfate Solution After Heating at 650 C.
at the tops of the bar graphs.

cent nitric acid-3 per cent hydrofluoric acid at 70 C, and boiling, acidified, copper sulfate solution. The latter solution contained 50 g of $CuSO_4 \cdot 5H_2O$, 50 ml of concentrated sulfuric acid, and 420 ml of water.

The nitric and nitric-hydrofluoric acid test specimens were $1\frac{1}{2}$ in. long by 1 in. wide by $\frac{1}{4}$ in. thick and were polished on all sides with No. 120 emery paper, whereas the boiling acidified copper sulfate solution test specimens consisted of unpolished, sandblasted strips 5 in. long by $\frac{1}{2}$ in. wide by $\frac{1}{16}$ in. thick.

The boiling 65 per cent nitric acid tests were conducted in accordance with the tentative ASTM Standard A 262.[5] The corrosion rates from the third 48-hr test period were used in evaluating the steels because it has been found that, in general, the third period rate is about equal to the average rate of five 48-hr test periods. A corrosion rate of 0.002-in. penetration per month was arbitrarily adopted as the limit of satisfactory corrosion resistance.

The nitric-hydrofluoric acid tests were conducted for a single period of a 1 hr

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[5] Tentative Recommended Practice for Boiling Nitric Acid Test for Corrosion-Resisting Steels (A 262 – 55 T), 1955 Book of ASTM Standards, Part 1, p. 1108.

TABLE II.—RESULTS OF BOILING 65 PER CENT NITRIC ACID TESTS ON SPECIMENS HEATED AT 650 C.

Heat	Composition, per cent						Third Period Corrosion Rate. Penetration, in. per month			
								Heated ½ hr at 1075 C and Air-Cooled		
	Chromium	Manganese	Nickel	Silicon	Carbon[a]	Nitrogen[a]	Heated ½ hr at 1075 C and Air-Cooled	Reheated 1 hr at 650 C	Reheated 6 hr at 650 C	Reheated 24 hr at 650 C
S318	18.25	7.0	4.5	0.4	0.017	0.22	N.T.[b]	N.T.	N.T.	N.T.
S319	18.25	7.0	4.5	0.4	0.020	0.22	N.T.	N.T.	N.T.	N.T.
S286	18.25	7.0	4.5	0.4	0.028	0.22	N.T.	N.T.	N.T.	N.T.
S287	18.25	7.0	4.5	0.4	0.030	0.21	N.T.	N.T.	N.T.	N.T.
S288	18.25	7.0	4.5	0.4	0.036	0.21	N.T.	N.T.	N.T.	N.T.
S289	18.25	7.0	4.5	0.4	0.040	0.22	N.T.	N.T.	N.T.	N.T.
S324	18.25	7.0	5.0	0.4	0.015	0.22	N.T.	N.T.	N.T.	N.T.
S325	18.25	7.0	5.0	0.4	0.024	0.22	0.00099	0.0010	0.00098	0.0013
S326	18.25	7.0	5.0	0.4	0.028	0.22	0.0010	0.0010	0.0010	0.0014
S327	18.25	7.0	5.0	0.4	0.029	0.22	0.00099	0.0010	0.0010	0.0012
S328	18.25	7.0	5.0	0.4	0.034	0.22	0.0010	0.0011	0.0019	0.016
S329	18.25	7.0	5.0	0.4	0.043	0.22	0.0010	0.0012	0.0020	0.048
S151	18.25	7.0	6.0	0.4	0.010	0.22	0.0008	0.00083	0.00096	0.00093
S152	18.25	7.0	6.0	0.4	0.021	0.23	0.00082	0.00082	0.00092	0.0012
S153	18.25	7.0	6.0	0.4	0.026	0.22	0.00082	0.00089	0.0011	0.0016
S154	18.25	7.0	6.0	0.4	0.028	0.22	0.00089	0.00086	0.0012	0.0036
S198	18.25	7.0	6.0	0.4	0.037	0.22	0.00094	0.00096	0.0011	0.0075
S155	18.25	9.0	4.0	0.4	0.013	0.20	0.0010	0.0011	0.0011	0.0011
S157	18.25	9.0	4.0	0.4	0.025	0.23	0.0011	0.0012	0.0013	0.0018
S158	18.25	9.0	4.0	0.4	0.027	0.22	0.0010	0.0011	0.0015	0.0022
S156	18.25	9.0	4.0	0.4	0.029	0.22	0.0012	0.0012	0.0016	0.0027
S311	18.25	9.0	4.0	0.4	0.037	0.22	0.0013	0.0014	0.0015	0.0020
S199	18.25	9.0	4.0	0.4	0.043	0.22	0.0013	0.0013	0.0061	0.037
S320	18.25	9.0	4.5	0.4	0.012	0.22	0.0011	0.0010	0.00098	0.0011
S321	18.25	9.0	4.5	0.4	0.023	0.22	0.0010	0.0010	0.00098	0.0011
S293	18.25	9.0	4.5	0.4	0.028	0.22	0.0013	0.0011	0.0012	0.0029
S292	18.25	9.0	4.5	0.4	0.034	0.21	0.0011	0.0010	0.0011	0.0014
S294	18.25	9.0	4.5	0.4	0.040	0.22	0.0011	0.0012	0.0018	0.0074
S159	18.25	9.0	5.0	0.4	0.010	0.22	0.0010	0.0010	0.0010	0.00097
S160	18.25	9.0	5.0	0.4	0.020	0.22	0.00093	0.00099	0.0012	0.0020
S161	18.25	9.0	5.0	0.4	0.021	0.21	0.00095	0.00099	0.0011	0.0012
S260	18.25	9.0	5.0	0.4	0.026	0.21	0.00099	0.0010	0.0010	0.0013
S200	18.25	9.0	5.0	0.4	0.035	0.23	0.0011	0.0011	0.0011	0.0038
S162	18.25	9.0	5.0	0.4	0.036	0.23	0.00094	0.0011	0.0022	0.071
S163	18.25	9.0	6.0	0.4	0.010	0.21	0.00078	0.00075	0.00080	0.00078
S164	18.25	9.0	6.0	0.4	0.021	0.22	0.00086	0.00085	0.00089	0.0011
S165	18.25	9.0	6.0	0.4	0.024	0.22	0.00095	0.00088	0.0010	0.0012
S166	18.25	9.0	6.0	0.4	0.027	0.22	0.0010	0.00094	0.020	0.13
S201	18.25	9.0	6.0	0.4	0.036	0.23	0.0011	0.0023	0.024	0.15
S167	18.25	9.0	7.0	0.4	0.015	0.22	0.00078	0.00073	0.00072	0.00073
S313	18.25	9.0	7.0	0.4	0.020	0.22	0.00073	0.00074	0.00079	0.0012
S169	18.25	9.0	7.0	0.4	0.028	0.23	0.00083	0.00078	0.0042	0.022
S170	18.25	9.0	7.0	0.4	0.034	0.22	0.00087	0.00087	0.059	0.11
S202	18.25	9.0	7.0	0.4	0.040	0.22	0.00081	0.0012	0.0065	0.11

TABLE II.—*Concluded.*

Heat	Composition, per cent						Third Period Corrosion Rate. Penetration, in. per month			
	Chromium	Manganese	Nickel	Silicon	Carbon[a]	Nitrogen[a]	Heated ½ hr at 1075 C and Air-Cooled	Heated ½ hr at 1075 C and Air-Cooled		
								Reheated 1 hr at 650 C	Reheated 6 hr at 650 C	Reheated 24 hr at 650 C
S171	18.25	9.0	10.0	0.4	0.015	0.21	0.00053	0.00055	0.00057	0.00058
S262	18.25	9.0	10.0	0.4	0.018	0.19	0.00054	0.00050	0.00055	0.0014
S173	18.25	9.0	10.0	0.4	0.025	0.22	0.00062	0.00064	0.014	0.078
S172	18.25	9.0	10.0	0.4	0.027	0.22	0.00057	0.00062	0.012	0.11
S174	18.25	9.0	10.0	0.4	0.033	0.23	0.00064	0.0026	0.061	0.14
S203	18.25	9.0	10.0	0.4	0.042	0.22	0.00069	0.0040	0.053	0.21
S322	18.25	11.0	4.5	0.4	0.019	0.22	0.0011	0.0011	0.0010	0.0012
S323	18.25	11.0	4.5	0.4	0.022	0.22	0.0011	0.0012	0.0011	0.0012
S298	18.25	11.0	4.5	0.4	0.026	0.22	0.0011	0.0011	0.0012	0.0014
S299	18.25	11.0	4.5	0.4	0.034	0.22	0.0010	0.0012	0.018	0.023
S300	18.25	11.0	4.5	0.4	0.039	0.22	0.0011	0.0014	0.0096	0.059
S301	18.25	11.0	4.5	0.4	0.04	0.21	0.0012	0.0034	0.027	0.086
S179	18.25	11.0	5.0	0.4	0.014	0.21	0.00094	0.0009	0.00093	0.0011
S180	18.25	11.0	5.0	0.4	0.022	0.23	0.0011	0.0010	0.0011	0.0015
S181	18.25	11.0	5.0	0.4	0.025	0.22	0.0010	0.0011	0.0013	0.019
S182	18.25	11.0	5.0	0.4	0.033	0.23	0.0011	0.0011	0.020	0.095
S205	18.25	11.0	5.0	0.4	0.035	0.23	0.0011	0.0016	0.083	0.15
S183	18.25	11.0	6.0	0.4	0.015	0.21	0.00079	0.00077	0.00077	0.00084
S184	18.25	11.0	6.0	0.4	0.023	0.24	0.00086	0.00086	0.00086	0.0041
S185	18.25	11.0	6.0	0.4	0.027	0.23	0.00088	0.00089	0.0059	0.055
S186	18.25	11.0	6.0	0.4	0.035	0.22	0.00090	0.0018	0.052	0.11
S206	18.25	11.0	6.0	0.4	0.04	0.23	0.00091	0.0017	0.057	0.11
S187	18.25	11.0	7.0	0.4	0.016	0.21	0.00066	0.00065	0.00069	0.00072
S265	18.25	11.0	7.0	0.4	0.021	0.20	0.00068	0.00069	0.00070	0.00069
S189	18.25	11.0	7.0	0.4	0.026	0.22	0.00081	0.0012	0.014	0.074
S188	18.25	11.0	7.0	0.4	0.029	0.23	0.00078	0.0008	0.035	0.076
S190	18.25	11.0	7.0	0.4	0.038	0.23	0.00084	0.010	0.078	0.15

[a] Actual content.

[b] N.T. = Not tested for intergranular corrosion resistance because of ingot porosity.

duration. The test was limited to a single period because of the high weight losses produced by this test solution, and because this solution is an extremely sensitive detector of intergranular corrosion. The susceptibility of the steels to intergranular corrosion was based on the ratio of the corrosion rate after heat-treatment to that in the solution-heat-treated condition, and the steel was considered to have failed intergranularly if this ratio exceeded about 1.25.

With the exceptions stated below, the boiling copper sulfate tests were made as designated in the tentative ASTM Standards A 393 - 55 T.[6] The ratio of volume of solution to specimen area was approximately 25 ml per sq in. instead of the recommended 50 ml per sq in. In order to more closely evaluate the resistance of the steels to intergranular corrosion, the specimens were bent through 180 deg and examined for cracks at a magnification of 50× after test periods of approximately 7, 24, 50, 100, 200, 300,

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[6] Tentative Recommended Practice for Conducting Acidified Copper Sulfate Test for Intergranular Attack in Austenitic Stainless Steel (A 393 - 55 T), 1955 Book of ASTM Standards, Part 1, p. 1135.

TABLE III.—RESULTS OF TESTS IN 10 PER CENT NITRIC-3 PER CENT HYDROFLUORIC ACID AT 70 C ON SPECIMENS HEATED AT 650 C.

Heat	Composition, per cent						Heated ½ hr at 1075 C and Air-Cooled	Heated ½ hr at 1075 C and Air-Cooled		
	Chromium	Manganese	Nickel	Silicon	Carbon[a]	Nitrogen[a]		Reheated 1 hr at 650 C	Reheated 6 hr at 650 C	Reheated 24 hr at 650 C
S318	18.25	7.0	4.5	0.4	0.017	0.22	N.T.[b]	N.T.	N.T.	N.T.
S319	18.25	7.0	4.5	0.4	0.020	0.22	N.T.	N.T.	N.T.	N.T.
S286	18.25	7.0	4.5	0.4	0.028	0.22	N.T.	N.T.	N.T.	N.T.
S287	18.25	7.0	4.5	0.4	0.030	0.21	N.T.	N.T.	N.T.	N.T.
S288	18.25	7.0	4.5	0.4	0.036	0.21	N.T.	N.T.	N.T.	N.T.
S289	18.25	7.0	4.5	0.4	0.040	0.22	N.T.	N.T.	N.T.	N.T.
S324	18.25	7.0	5.0	0.4	0.015	0.22	N.T.	N.T.	N.T.	N.T.
S325	18.25	7.0	5.0	0.4	0.024	0.22	0.94	0.98	0.94	1.08
S326	18.25	7.0	5.0	0.4	0.028	0.22	1.15	1.22	1.26	1.49
S327	18.25	7.0	5.0	0.4	0.029	0.22	1.04	1.23	1.12	1.46
S328	18.25	7.0	5.0	0.4	0.034	0.22	1.17	1.31	1.38	5.03
S329	18.25	7.0	5.0	0.4	0.043	0.22	1.22	1.38	2.42	6.14
S151	18.25	7.0	6.0	0.4	0.010	0.22	0.86	0.88	0.82	1.08
S152	18.25	7.0	6.0	0.4	0.021	0.23	0.72	0.78	0.81	3.43
S153	18.25	7.0	6.0	0.4	0.026	0.22	0.82	0.91	0.94	3.26
S154	18.25	7.0	6.0	0.4	0.028	0.22	0.91	0.96	0.92	4.38
S198	18.25	7.0	6.0	0.4	0.037	0.22	1.46	1.18	1.09	3.38
S155	18.25	9.0	4.0	0.4	0.013	0.20	1.08	1.09	1.06	1.19
S157	18.25	9.0	4.0	0.4	0.025	0.23	1.08	1.22	1.15	1.40
S158	18.25	9.0	4.0	0.4	0.027	0.22	1.27	1.44	1.70	4.36
S156	18.25	9.0	4.0	0.4	0.029	0.22	0.96	1.17	2.14	4.98
S311	18.25	9.0	4.0	0.4	0.037	0.22	1.36	1.52	1.43	3.72
S199	18.25	9.0	4.0	0.4	0.043	0.22	2.00	1.74	3.00	7.75
S320	18.25	9.0	4.5	0.4	0.012	0.22	1.16	1.29	1.06	1.03
S321	18.25	9.0	4.5	0.4	0.023	0.22	1.28	1.36	1.12	1.10
S293	18.25	9.0	4.5	0.4	0.028	0.22	1.25	1.09	1.38	2.48
S292	18.25	9.0	4.5	0.4	0.034	0.21	1.56	1.51	1.84	4.20
S294	18.25	9.0	4.5	0.4	0.040	0.22	1.30	1.10	1.44	3.52
S159	18.25	9.0	5.0	0.4	0.010	0.22	1.20	1.25	1.03	0.85
S160	18.25	9.0	5.0	0.4	0.020	0.22	0.90	1.03	1.24	2.13
S161	18.25	9.0	5.0	0.4	0.021	0.21	1.06	1.12	1.03	1.14
S260	18.25	9.0	5.0	0.4	0.026	0.21	1.40	1.39	1.09	1.62
S200	18.25	9.0	5.0	0.4	0.035	0.23	1.67	1.44	1.58	2.69
S162	18.25	9.0	5.0	0.4	0.036	0.23	1.11	1.36	1.77	6.16
S163	18.25	9.0	6.0	0.4	0.010	0.21	0.89	0.96	0.82	0.85
S164	18.25	9.0	6.0	0.4	0.021	0.22	0.84	0.90	0.89	1.85
S165	18.25	9.0	6.0	0.4	0.024	0.22	0.97	1.04	1.16	3.87
S166	18.25	9.0	6.0	0.4	0.025	0.22	1.08	2.02	2.29	3.47
S201	18.25	9.0	6.0	0.4	0.036	0.23	1.32	1.56	3.77	4.19
S167	18.25	9.0	7.0	0.4	0.015	0.22	0.85	0.90	0.76	0.79
S313	18.25	9.0	7.0	0.4	0.020	0.22	0.92	1.18	0.83	1.10
S169	18.25	9.0	7.0	0.4	0.028	0.23	0.78	1.05	2.23	3.63
S170	18.25	9.0	7.0	0.4	0.034	0.22	0.96	2.41	4.82	5.46
S202	18.25	9.0	7.0	0.4	0.040	0.22	1.21	1.25	2.82	4.75

TABLE III.—*Concluded.*

Heat	Composition, per cent						Heated ½ hr at 1075 C and Air-Cooled	Heated ½ hr at 1075 C and Air-Cooled		
	Chromium	Manganese	Nickel	Silicon	Carbon[a]	Nitrogen[a]		Reheated 1 hr at 650 C	Reheated 6 hr at 650 C	Reheated 24 hr at 650 C
S171	18.25	9.0	10.0	0.4	0.015	0.21	0.62	0.67	0.55	0.77
S262	18.25	9.0	10.0	0.4	0.018	0.19	0.75	0.83	0.62	1.12
S173	18.25	9.0	10.0	0.4	0.025	0.22	0.66	0.84	2.25	3.47
S172	18.25	9.0	10.0	0.4	0.027	0.22	0.58	0.95	2.12	3.63
S174	18.25	9.0	10.0	0.4	0.033	0.23	0.57	2.15	2.27	3.02
S203	18.25	9.0	10.0	0.4	0.042	0.22	0.96	1.53	3.05	3.71
S322	18.25	11.0	4.5	0.4	0.019	0.22	1.21	1.42	1.28	1.44
S323	18.25	11.0	4.5	0.4	0.022	0.22	1.44	1.68	1.52	1.85
S298	18.25	11.0	4.5	0.4	0.026	0.22	1.56	1.61	1.60	1.62
S299	18.25	11.0	4.5	0.4	0.034	0.22	1.50	1.79	2.50	4.20
S300	18.25	11.0	4.5	0.4	0.039	0.22	1.50	1.64	2.22	5.10
S301	18.25	11.0	4.5	0.4	0.04	0.21	1.40	1.85	3.45	4.90
S179	18.25	11.0	5.0	0.4	0.014	0.21	1.11	1.06	1.02	1.09
S180	18.25	11.0	5.0	0.4	0.022	0.23	1.08	1.07	1.46	3.43
S181	18.25	11.0	5.0	0.4	0.025	0.22	1.13	1.18	1.80	6.81
S182	18.25	11.0	5.0	0.4	0.033	0.23	1.26	3.01	4.14	6.75
S205	18.25	11.0	5.0	0.4	0.035	0.23	1.57	2.68	5.1	10.8
S183	18.25	11.0	6.0	0.4	0.015	0.21	0.94	0.90	0.88	0.96
S184	18.25	11.0	6.0	0.4	0.023	0.24	0.98	0.88	0.88	1.51
S185	18.25	11.0	6.0	0.4	0.027	0.23	1.11	1.10	1.87	5.37
S186	18.25	11.0	6.0	0.4	0.035	0.22	1.37	2.01	4.65	6.99
S206	18.25	11.0	6.0	0.4	0.04	0.23	1.51	2.50	4.25	8.16
S187	18.25	11.0	7.0	0.4	0.016	0.21	0.90	0.76	0.75	0.67
S265	18.25	11.0	7.0	0.4	0.021	0.20	1.13	1.30	0.91	0.85
S189	18.25	11.0	7.0	0.4	0.026	0.22	1.14	1.04	3.13	3.39
S188	18.25	11.0	7.0	0.4	0.029	0.23	0.86	1.19	1.95	2.26
S190	18.25	11.0	7.0	0.4	0.038	0.23	1.17	1.79	3.89	6.14

[a] Actual content.

[b] N.T. = Not tested for intergranular corrosion resistance because of ingot porosity.

and 500 hr. The specimens were removed from test either when severe cracks were observed or at the end of 500 hr.

DISCUSSION

Analysis of the intergranular corrosion data, summarized in Figs. 3 to 5 and in Tables II and III, revealed that the maximum carbon content at which the steel retains its immunity under the conditions of test is related to the sum of the manganese and nickel contents of the steel. This relationship between the carbon limit and the alloy content of the steel is similar to the relationship which exists in the 0.03 per cent carbon austenitic chromium-nickel steels (**2**).

To illustrate this relationship, the carbon limit derived from tests in boiling, 65 per cent nitric acid solution on specimens heated 6 hr at 650 C is plotted in Fig. 6 as a function of the sum of the manganese-plus-nickel contents of the steel. It is seen that the carbon limit decreases as the sum of these elements increases. In view of the commercial importance of developing steels exhibiting the maximum tolerance for carbon, the relationship between carbon limit and the manganese-plus-nickel content of the steel was evaluated for each test condition. The results of this study are discussed below in greater detail.

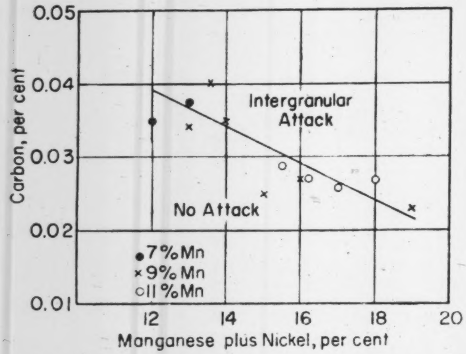

FIG. 6.—Relationship Between Carbon Limit for Immunity to Intergranular Corrosion After 6 hr at 650 C and the Manganese-Plus-Nickel Content of the Steel Determined from Tests in Boiling 65 per cent Nitric Acid.

The carbon limits for 1, 6, and 24 hr at 650 C are tabulated below:

Heating Time at 650 C, hr	Manganese + Nickel, per cent	Carbon Limit, per cent		
		Boiling 65 per cent HNO_3 Acid	10 per cent HNO_3-3 per cent HF at 70 C	Boiling Acidified $CuSO_4$ Solution
1.........	14	0.050	0.040	0.050
1.........	18	0.035	0.030	0.040
6.........	14	0.035	0.030	0.030
6.........	18	0.025	0.020	0.020
24.........	14	0.025	0.020	0.020
24.........	18	0.020	0.015	0.015

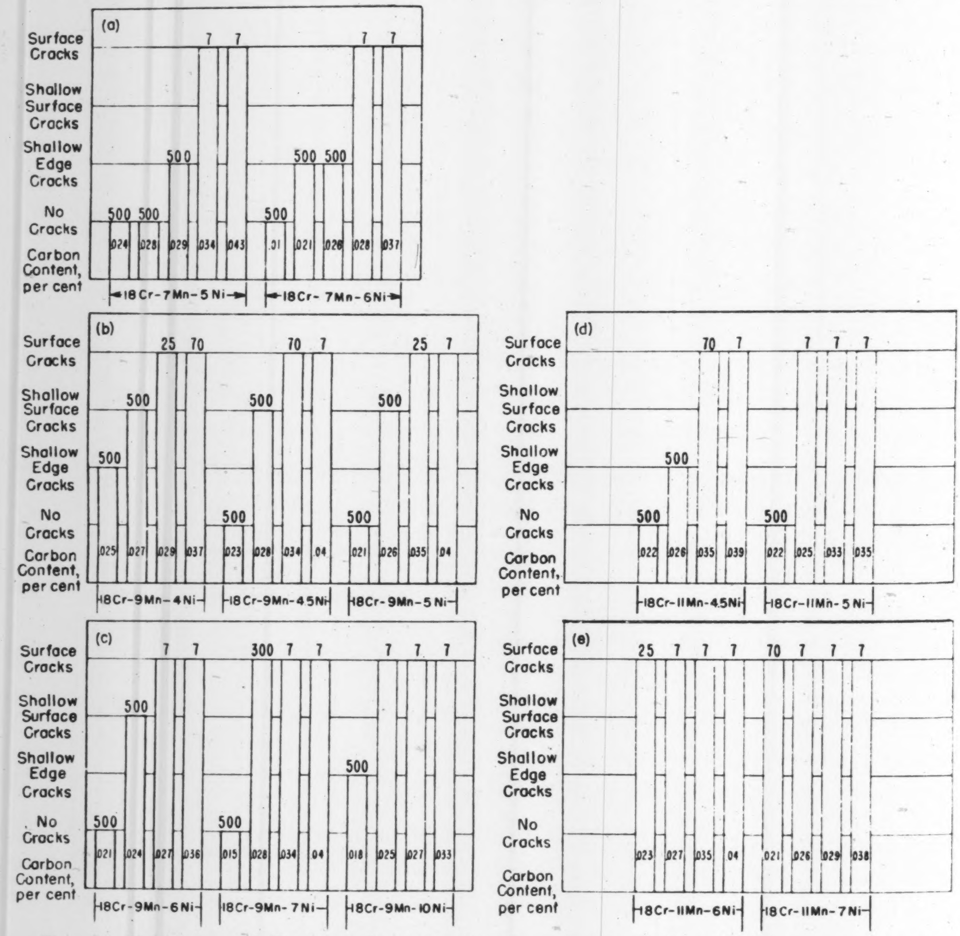

FIG. 7.—Resistance of Steels to Intergranular Corrosion in Boiling Acidified Copper Sulfate Solution After Heating 100 hr at 550 C.

The immersion times are shown at the tops of the bar graphs.

In each test condition, the carbon limits decrease as the manganese-plus-nickel increases. The carbon limits based on the tests in nitric acid are in reasonably good agreement with those derived from the tests in boiling-acidified-copper-sulfate solution. Carbon limits for partial immunity based on the nitric-hydrofluoric acid test are slightly lower, reflecting the greater severity of the latter test solution. Since current commercial arc-furnace melting practice restricts the minimum carbon content to about 0.025 per cent, it is evident that complete immunity to intergranular attack, as indicated by resistance after 24 hr at 650 C, imposes a limit of 14 per cent on the manganese-plus-nickel contents. Somewhat higher percentages of these elements are permissible if the heating time is limited to 1 hr at 650 C.

EFFECT OF EXTENDED HEATING AT 300 TO 550 C

The corrosion tests conducted on specimens heated 16 weeks at 300 C, 8 weeks at 400 C, and 100 hr at 550 C were limited to boiling 65 per cent nitric acid and boiling acidified copper sulfate solutions.

None of the steels exhibited intergranular attack after heating at 300 C for 16 weeks or at 400 C for 8 weeks. As shown in Table I and in Fig. 7, steels containing 0.025 per cent carbon were resistant to attack after 100 hr at 550 C provided the total manganese plus nickel was less than 14 per cent. These results are in satisfactory agreement with the predictions for complete immunity based on the 650 C tests.

SUMMARY AND CONCLUSIONS

The effect of up to 0.045 per cent carbon and the sum of the manganese and nickel contents on the intergranular corrosion resistance of austenitic steels containing 18 per cent chromium, 7 to 11 per cent manganese, 4 to 10 per cent nickel, and 0.22 per cent nitrogen was investigated. With one exception, the study was restricted to steels containing more manganese than nickel.

The carbon tolerance was found to decrease as the sum of the manganese-plus-nickel increased above 14 per cent. Steels containing about 0.05 per cent carbon and up to 14 per cent manganese-plus-nickel, and about 0.035 per cent carbon and 18 per cent manganese-plus-nickel are resistant to intergranular attack after 1 hr at 650 C. Those containing 0.02 to 0.025 per cent carbon and 14 per cent manganese-plus-nickel and about 0.02 per cent carbon and 18 per cent manganese-plus-nickel are resistant after 24 hr at 650 C. Complete immunity as indicated by the resistance of the steels after heating 24 hr at 650 C, imposes a limit of 14 per cent manganese-plus-nickel since current arc-melting practice limits carbon to about 0.025 per cent minimum. The economics of melting also suggest that a similar manganese-plus-nickel limit should be applied to the steels meeting the requirements after 1 hr at 650 C, although the carbon limits for partial immunity are less restrictive than for complete immunity.

The steels meeting the requirements for 1 hr at 650 C should be satisfactory for use in the as-welded or stress-relieved conditions in a great majority of applications, and those meeting the requirements for 24 hr at 650 C should be satisfactory for continuous service at temperatures in the carbide precipitation temperature range. Although limited to short periods of exposure at higher temperatures, the steels meeting the requirements for 1 hr at 650 C can be heated continuously at temperatures as high as 400 C without danger of becoming susceptible to intergranular attack.

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(2) W. O. Binder, C. M. Brown, and R. Franks, "Resistance to Sensitization of Austenitic Chromium-Nickel Stainless Steels of 0.03 Per Cent Max Carbon Content," *Transactions*, Am. Soc. Metals, Vol. 41, p. 1301 (1949).

(3) Symposium on Evaluation Tests for Stainless Steels, Am. Soc. Testing Mats. (1949). Issued as separate publication *ASTM STP No. 93.*

(4) R. Franks, W. O. Binder, and J. Thompson, "Austenitic Chromium-Manganese-Nickel Steels Containing Nitrogen," *Transactions*, Am. Soc. Metals, Vol. 47, p. 231 (1955).

DISCUSSION

MR. W. G. RENSHAW[1] (*presented in written form*).—The authors are to be congratulated on their very complete investigation of the influence of composition on intergranular corrosion resistance of these steels. The results provide very valuable information regarding resistance to intergranular corrosion where these new steels are to be welded or heated short times in the sensitizing range.

Mr. Binder and his coworkers have shown that within the limit of 14 per cent manganese plus nickel content, a 0.025 per cent carbon content will assure complete resistance to intergranular attack after heating 24 hr at 650 C. Based on this they believe that a steel meeting this requirement should be satisfactory for continuous service at temperatures in the carbide precipitation temperature range. We are wondering whether resistance to intergranular attack after 24 hr at 650 C can be considered sufficient assurance that a material is satisfactory for continuous service at this temperature. Kiefer and Sheridan[2] in their discussion of low-carbon austenitic chromium-nickel stainless steels suggest that a more conservative 100 hr at 650 C (1200 F) may only be a fair indication of resistance to intergranular corrosion after longer periods of exposure. They caution, however, that even this test may not be positive proof since there is a possibility that some failures might have resulted after longer time in the sensitizing range or longer periods of time in the test medium. While nitrogen in the amounts added to these chromium-nickel-manganese steels has not shown any apparent promotion of intergranular corrosion after short sensitizing time, we wonder whether this is still true for prolonged exposure perhaps as long as 500 or 1000 hr at 550 C (1000 F) or 650 C (1200 F).

In regard to the authors' statement that the third period corrosion rate is usually about the same as the average of five periods in the boiling nitric acid test, we agree this may be frequently true provided a certain standard grain size is employed. However, where variation in grain size exists, two identical compositions can behave quite differently, since the number of grains which fall out of the test specimen and the ease with which this occurs can influence corrosion rates seriously. This grain fall out may not be significant in many instances until the fourth or fifth 48-hr period, while in others it may occur after one or two. For this reason we believe no concise rule can be set up which can establish a relation between the average (of five) and third period corrosion rates. This is, however, a rather useful time saving assumption, which is fairly applicable in cases where little or no intergranular attack is encountered.

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[1] Allegheny Ludlum Steel Corp., Research Lab., Brackenridge, Pa.

[2] George C. Kiefer and C. M. Sheridan, "Effect of Composition on Low Carbon Austenitic Chromium-Nickel Stainless Steels," *Yearbook*, Am. Iron and Steel Inst., pp. 476–499 (1948).

MR. J. J. HEGER.[3]—It is interesting to note that the carbon and nickel limits for porosity shown in Fig. 1 and the carbon and nickel limits for delta ferrite shown in Fig. 2 are quite similar. Thus one wonders if the porosity is controlled by the carbon and nickel or by the delta ferrite.

The carbon limits for resistance to intergranular corrosion by nitric acid are higher than the carbon limits for intergranular corrosion by copper sulfate sulfuric acid. Does this mean that the copper sulfate test is somewhat more aggressive than the nitric acid test?

The authors used a corrosion rate at the end of three periods, rather than the average corrosion rate after five periods. Is there evidence that the corrosion rate at the end of three periods is about the same as the average corrosion rate after five periods?

MR. J. THOMPSON (*author's closure*).—The authors wish to thank Mr. Renshaw for his discussion of this paper.

Evaluation of the steels for intergranular corrosion resistance after prolonged periods within the sensitizing range is desirable. While the tests made after heating the steels 24 hr at 650 C served to demonstrate that steels of certain compositions were highly resistant to intergranular attack, final evaluation could best be established under actual service conditions.

Although the presence of nitrogen appears to have no major influence on the promotion of intergranular corrosion after exposure of the steels for 100 hr within the carbide precipitation range, there are no data at this time to establish the effect of nitrogen after periods of 500 to 1000 hr at these temperatures.

Mr. Renshaw's comments regarding the influence of grain size on the corrosion rates of the steels after the third and fifth test periods in nitric acid are interesting. However, metallographic examination of these steels revealed no wide variation in grain size. The choice made of the rates from the third test period was based on experience from tests on a large number of steels in nitric acid for five 48-hr periods. Although, as noted by Mr. Renshaw, closest correlation was obtained on the steels having the highest resistance to intergranular attack, with few exceptions, the rates from the third period predicted with reasonable accuracy the values obtained from the average of five test periods.

In reply to Mr. Heger's comment regarding the relationship between the delta ferrite content and porosity of the steels shown in Figs. 1 and 2, it is believed that porosity is primarily influenced by the over-all composition rather than structure. For example, the 7 per cent manganese steels exhibiting porosity contained about the same amount of delta ferrite as the 9 and 11 per cent manganese steels of otherwise similar composition which were free from porosity.

The minor differences in carbon limits shown for the steels in the copper sulfate and nitric acid solutions might be attributed to the qualitative nature of the copper sulfate test as opposed to the more quantitative results of the boiling 65 per cent nitric acid solution. In general, the carbon limits based on the results of the tests in both solutions were in relatively good agreement. However, as stated by Mr. Heger, the copper sulfate test appears to impose slightly lower carbon limits than the boiling 65 per cent nitric acid test.

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[3] Chief Research Engineer—Stainless Steel, U. S. Steel Corp., Pittsburgh, Pa.

The reason for using the third period corrosion rate rather than the average of five periods is mentioned in reply to Mr. Renshaw's comments.

THE MECHANICAL PROPERTIES OF TYPE 201 CHROMIUM-NICKEL-MANGANESE STAINLESS STEEL SHEET*

By R. A. Walsh,[1] R. L. Cook,[1] and R. A. Lula[1]

Synopsis

The tensile and compressive properties were determined for two commercial heats of type 201 sheet with cold reductions from 0 to 60 per cent. The tension tests were made on standard specimens taken parallel and perpendicular to the rolling direction. The compression tests were made using the Montgomery-Templin compression jig.

The influence of stress-relieving heat treatments at 600 F, 800 F, and 900 F was determined for both tensile and compressive properties.

The results are presented as ultimate tensile strength, 0.2 per cent offset yield strength, elongation in 2 in., initial and tangent modulus, and stress-strain curves.

The object of this paper is to provide data that can be used for the design of those structures where type 201 is expected to find increased application.

AISI type 201 is a chromium-nickel-manganese austenitic stainless steel which has been recently standardized, and the following composition limits have been established:

Carbon, max per cent	0.15
Manganese, per cent	5.50 to 7.50
Chromium, per cent	16.00 to 18.00
Nickel, per cent	3.50 to 5.50
Nitrogen, max per cent	0.25

The various elements in this steel are so balanced that a relatively unstable microstructure results. As a consequence, a partial transformation to martensite occurs during cold rolling, resulting in high mechanical strength. In this respect, type 201 very much resembles type 301 chromium-nickel steel and, in fact, can be considered an alternate to type 301. Moreover, the resistance of type 201 in the mildly corrosive environments normally encountered in structural applications is also similar to that of type 301.

Since this newer steel requires only half as much nickel as type 301 but has very similar properties, it is expected to gain importance and to find widespread application in stationary facilities and especially in mobile structures where a high strength-weight ratio is required.

The object of this paper is to present a detailed investigation of the tensile and compressive properties of type 201 in both the annealed and cold-rolled conditions. The data summarized here will be useful, it is hoped, in the solution of design and other problems involving consideration of mechanical properties.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] Allegheny Ludlum Steel Corp., Research Laboratory, Brackenridge, Pa.

EXPERIMENTAL PROCEDURE

Tension Tests:

Standard ASTM specimens 0.5 in. wide,[2] machined to accommodate a 2 in. gage length, were used for all tensile work. Two specimens were cut both longitudinal and transverse to the rolling direction. When agreement was considered not within acceptable limits, a new set of two specimens was used.

TABLE I.—MATERIALS

CHEMICAL COMPOSITION, PER CENT

Heat	C	Mn	P	S	Si	Cr	Ni	N_2	Cu
No. 25312	0.099	6.17	0.022	0.006	0.76	16.83	3.83	0.14	0.11
No. 69346	0.11	6.48	0.025	0.011	0.54	17.92	4.31	0.18	0.17

PRIOR PROCESSING AND METALLOGRAPHY

Heat	Initial Thickness, in.	Annealing		ASTM Grain Size	ASTM Inclusion Rating
		Temperature, deg. Fahr.	Time, min.		
No. 25312	0.063	2000	4—ac[a]	No. 6	No. 2 Thin Series
No. 69346	0.066	2000	4—ac	No. 5–6	No. 2 Thin Series
No. 69346	0.122	2000	7—ac	No. 5–6	No. 2 Thin Series

[a] ac—Air cooled.

The tests were performed with one 400,000-lb capacity and two 120,000-lb capacity Baldwin-Southwark Universal testing machines. All three were calibrated and certified in May, 1955.

The strain was measured with a Model TIM microformer extensometer and was recorded on a Baldwin Model MAI autographic recorder.

The yield strength was determined by the 0.2 per cent offset method, and throughout this paper the term yield strength will be used to mean the stress at 0.2 per cent offset.

A constant strain rate of 0.005 in. per in. per min was used up to the yield point, and the rate of loading did not exceed 100,000 psi per min. From yield to failure, a crosshead speed of 0.5 in. per min was used. The rate of loading was determined by use of the Baldwin time interval marker integrated with the autographic recorder.

Compression Tests:

Compression tests were made by use of the Montgomery-Templin compression jig. Specimens were 2.625 in. long and 0.625 in. wide and were cut in the same manner as the tension specimens. Again, a new set of two specimens was tested if agreement between the two originals was considered inadequate.

The tests were performed on the two 120,000-lb capacity Baldwin-Southwark

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[2] Methods of Tension Testing of Metallic Materials (E 8 - 54 T), 1955 Book of ASTM Standards, Part I, p. 551.

Universal testing machines previously mentioned.

The strain was measured with a Baldwin-Lima-Hamilton Model PC5M-11 compressometer and was recorded on a Baldwin Model MAI autographic recorder.

A controlled crosshead speed of 0.02 in. per min was maintained for all compression testing.

Materials

The steel used in this investigation was taken from two production heats which had been processed on a continuous (tandem) hot rolling mill and then cold rolled to the initial thicknesses shown in Table I. The material was subsequently mill annealed at 2000 F. In order to avoid inducing any directional properties, no leveling operation was used. The compositions of the two heats are shown in Table I together with pertinent processing and metallographic data.

The various cold reductions (expressed as per cent decrease in thickness) were obtained by rolling on a single stand, four-high mill with no tension on the steel, using mill practice for sheet material. The cold reduction program is summarized in Table II. Also listed in this table are the amounts of martensite formed, as indicated by the Magne-Gage test for each cold reduction. This illustrates rather clearly the difference between the two heats in austenite stability which was the basis for their selection. Heat No. 25312 has lower carbon, manganese, chromium, nickel, and nitrogen than heat No. 69346 and is composed, therefore, of a less stable austenite. While no claim of accuracy is made for magnetic pull measurements on thin sheets, such tests are considered sufficiently good to illustrate the important differences in the amount of martensite formed upon cold rolling the two heats. For instance, heat No. 25312 forms about 17.5 per cent martensite when cold reduced about 40 per cent, while heat No. 69346 forms only 1.5 per cent martensite for the same cold reduction.

Note that heat No. 69346 was used in two thicknesses, one to match that of heat No. 25312 for more accurate comparison; one heavier gage to determine whether thickness, *per se*, had any effect on the mechanical characteristics for a given analysis.

TABLE II.—COLD REDUCTION SCHEDULE.

Heat	Cold Reduction, per cent	Gage Thickness After Reduction, in.	Martensite, per cent
No. 25312	0	0.063	<0.5
	5	0.060	0.6
	10	0.057	1.5
	15	0.054	4.8
	18	0.052	5.2
	27	0.046	12.2
	36	0.041	17.5
No. 69346	0	0.122	<0.5
	5	0.116	<0.5
	10	0.110	<0.5
	14	0.105	<0.5
	19	0.099	0.65
	28	0.089	1.15
	39	0.075	2.35
	58	0.051	8.95
No. 69346	0	0.066	<0.5
	5	0.063	<0.5
	10	0.060	<0.5
	15	0.056	<0.5
	21	0.052	0.6
	29	0.047	0.8
	40	0.040	1.5

Modulus Determination

The initial Young's modulus was obtained graphically from the slope of the tangent to the stress-strain curve at zero stress. The agreement between specimens in the same condition was good.

In order to determine the tangent modulus, the method outlined by Watter and Lincoln[3] was utilized. A generalized equation can be written for the stress-strain relationship for stainless steel:

$$e = \frac{S}{E_o} + K(S/E_o)^n \ldots\ldots\ldots (1)$$

or at the yield point:

$$e_{yp} = \frac{S_{yp}}{E_o} + K\left(\frac{S_{yp}}{E_o}\right)^n \ldots\ldots\ldots (2)$$

where:

S = stress, psi,

S_{yp} = stress at 0.2 per cent offset, psi,

e = strain, in. per in.,

E_o = Young's modulus at zero stress, psi, and

n = constant for each curve.

This equation can be solved for e (for 0.2 per cent offset yield method)

$$e = 0.002(S/S_{yp})^n + S/E_o \ldots\ldots (3)$$

The n value can be determined for each curve from the relationship

$$n = 1 + \frac{\log 17/7}{\log S_1/S_2} \ldots\ldots\ldots (4)$$

where S_1 is the secant yield stress determined by the intersection of the stress-strain curve with a line through the origin with a slope of $0.7E_o$, and S_2 is the secant yield stress for $0.85E_o$.

Curves calculated by the method outlined above have been checked with the experimental curves. Agreement was exceptionally good from zero stress to the 0.2 per cent offset yield. Differentiating Eq 3 gives

$$\frac{ds}{de} = E_t = \frac{S_{yp}}{A(S/S_{yp})^{n-1} + S_{yp}/E_o}$$

where $A = 0.002\,n$ and E_t is the tangent or instantaneous modulus.

TEST RESULTS

Longitudinal Versus Transverse Properties:

The results of the tension and compression tests in the longitudinal and transverse directions are shown in Tables III through V for annealed, cold-rolled, and stress-relieved specimens.

The stress-strain curves have been plotted for Heat No. 25312 only and are presented in Figs. 1 through 8. The stress-strain curves for annealed sheet in the longitudinal and transverse directions almost superpose. Hence, yield strengths are essentially the same, as are ultimate tensile strengths. However, the ordinates of the compressive stress-strain curves are higher than the corresponding ordinates of the tensile curves for both the longitudinal and transverse specimens. Consequently, yield strengths are higher in compression than in tension.

Upon cold rolling, these observations must be modified. Figures 9 through 15 are plots of strength *versus* per cent cold reduction. The average of two tests has been used in constructing these graphs.

Analysis of the tensile properties in the longitudinal and transverse directions show practically no difference for heat No. 25312, the less stable material. As indicated in Fig. 9, the longitudinal

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[3] M. Watter and R. A. Lincoln, "Strength of Stainless Steel Structural Members as a Function of Design," Allegheny Ludlum Steel Corp., Pittsburgh, Pa.

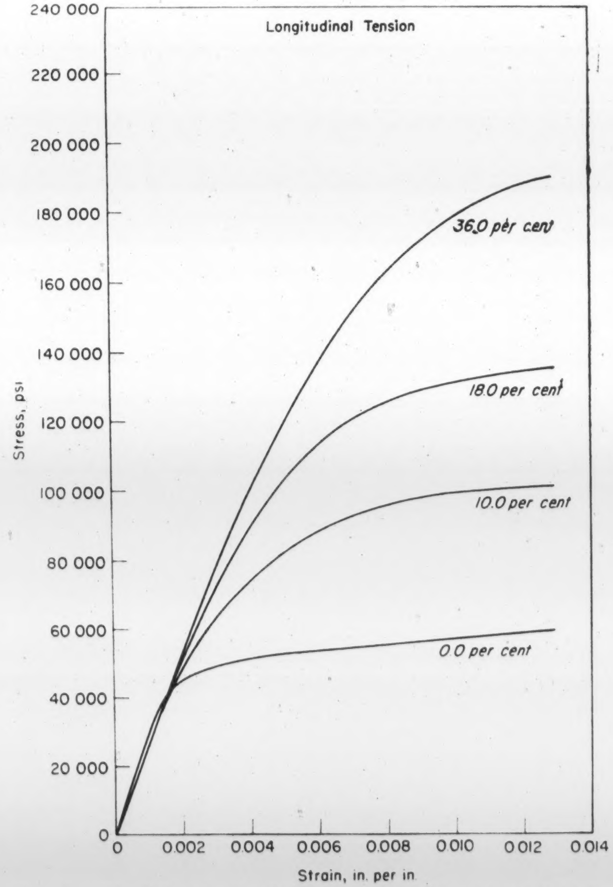

FIG. 1.—The Effect on the Stress-Strain Relationship of Cold Reducing 10 per cent, 18 per cent, and 36 per cent. Heat 25312.

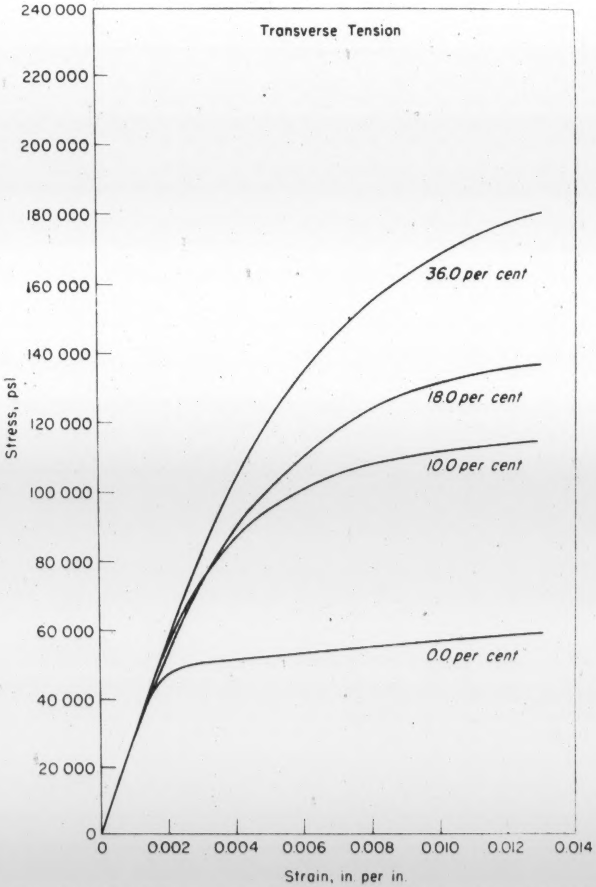

FIG. 2.—The Effect on the Stress-Strain Relationship of Cold Reducing 10 per cent, 18 per cent, and 36 per cent. Heat 25312.

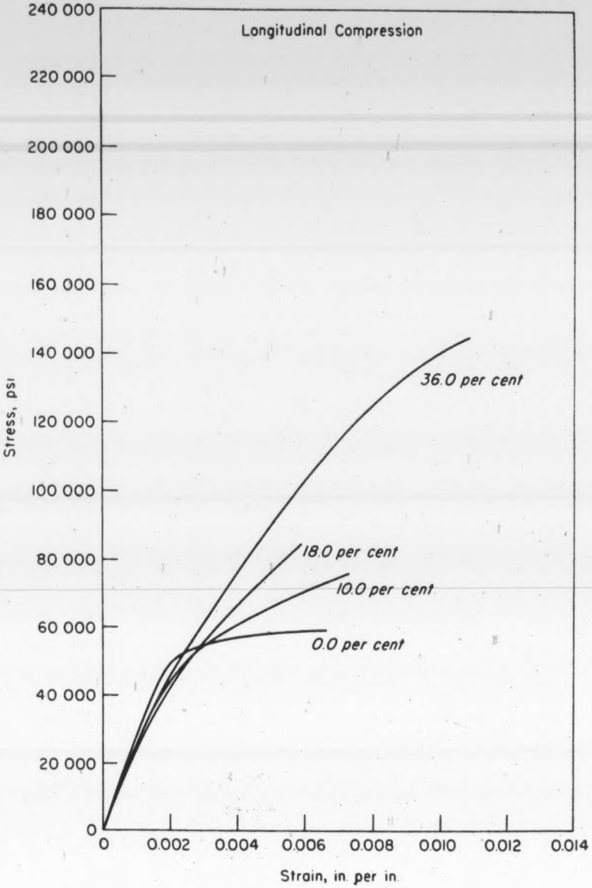

FIG. 3.—The Effect on the Stress-Strain Relationship of Cold Reducing 10 per cent, 18 per cent, and 36 per cent. Heat 25312.

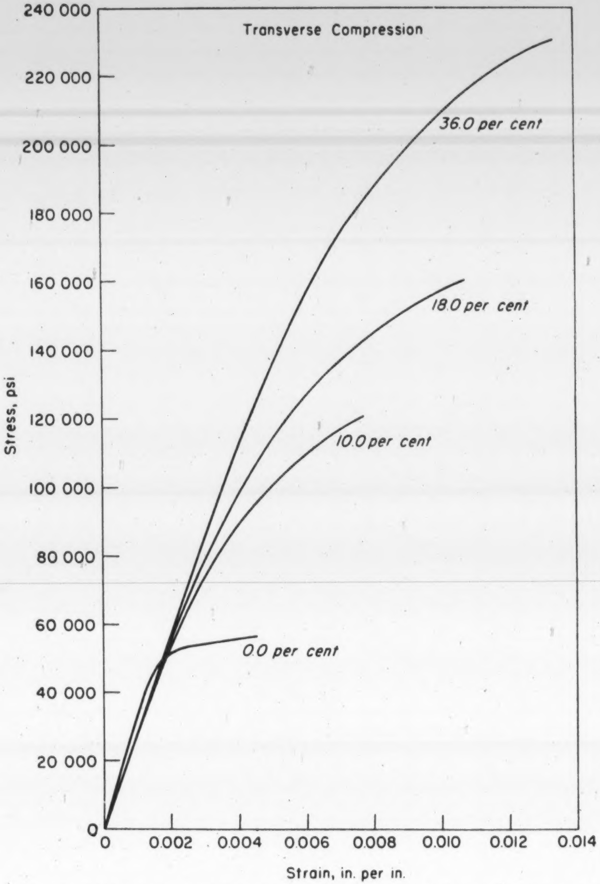

FIG. 4.—The Effect of the Stress-Strain Relationship of Cold Reducing 10 per cent, 18 per cent, and 36 per cent. Heat 25312.

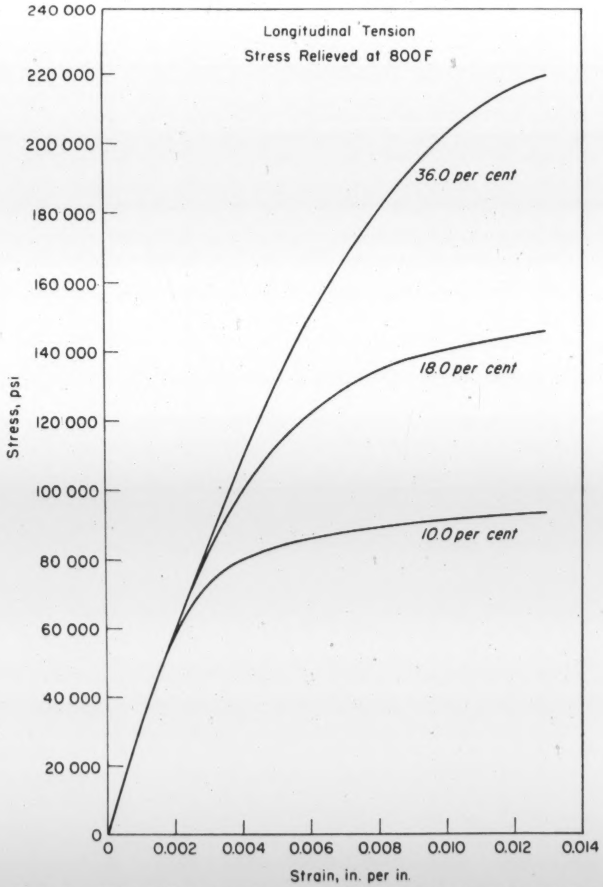

Fig. 5.—The Effect on the Stress-Strain Relationship of Stress Relieving for 12 hr after Cold Reductions of 10 per cent, 18 per cent, and 36 per cent. Heat 25312.

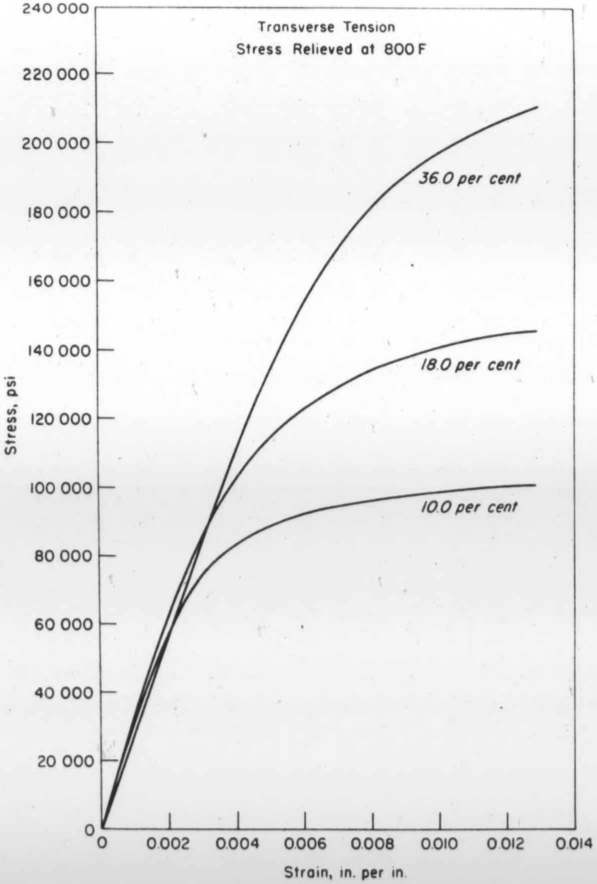

Fig. 6.—The Effect on the Stress-Strain Relationship of Stress Relieving for 12 hr after Cold Reductions of 10 per cent, 18 per cent, and 36 per cent. Heat 25312.

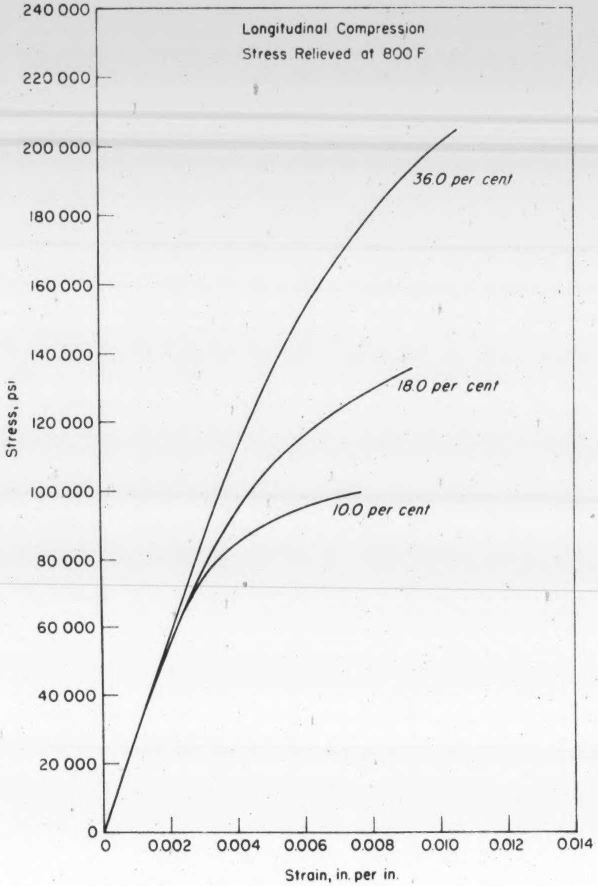

FIG. 7.—The Effect on the Stress-Strain Relationship of Stress Relieving for 12 hr after Cold Reductions of 10 per cent, 18 per cent and 36 per cent. Heat 25312.

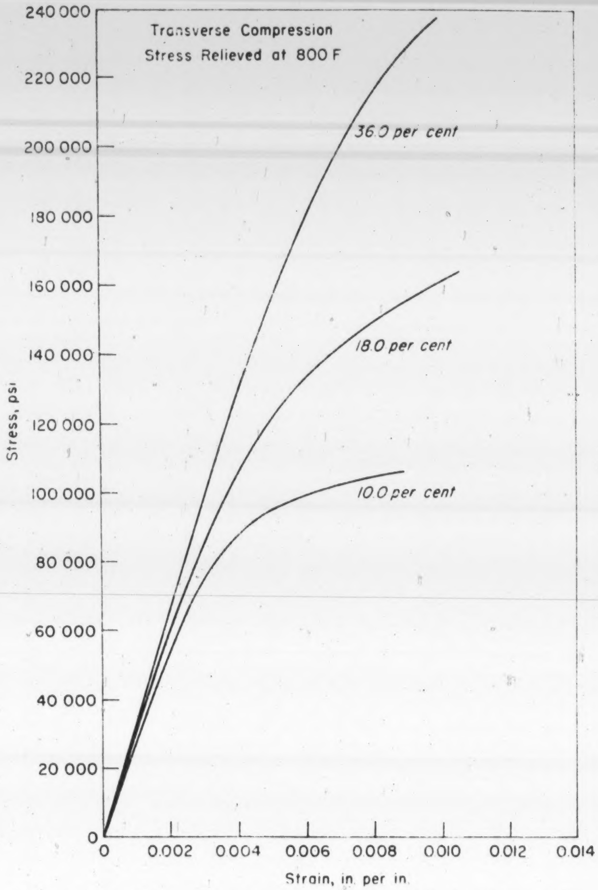

FIG. 8.—The Effect on the Stress-Strain Relationship of Stress Relieving for 12 hr after Cold Reductions of 10 per cent, 18 per cent, and 36 per cent. Heat 25312.

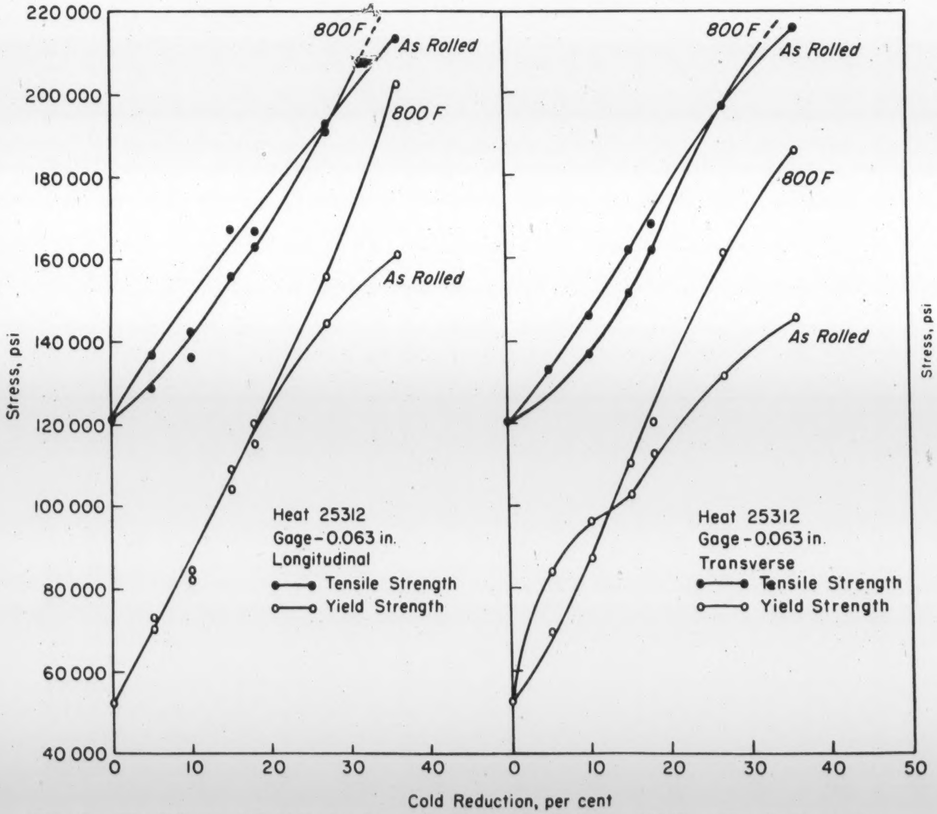

FIG. 9.—The Effects of Cold Reduction and of Stress Relieving at 800 F on Tensile Properties.

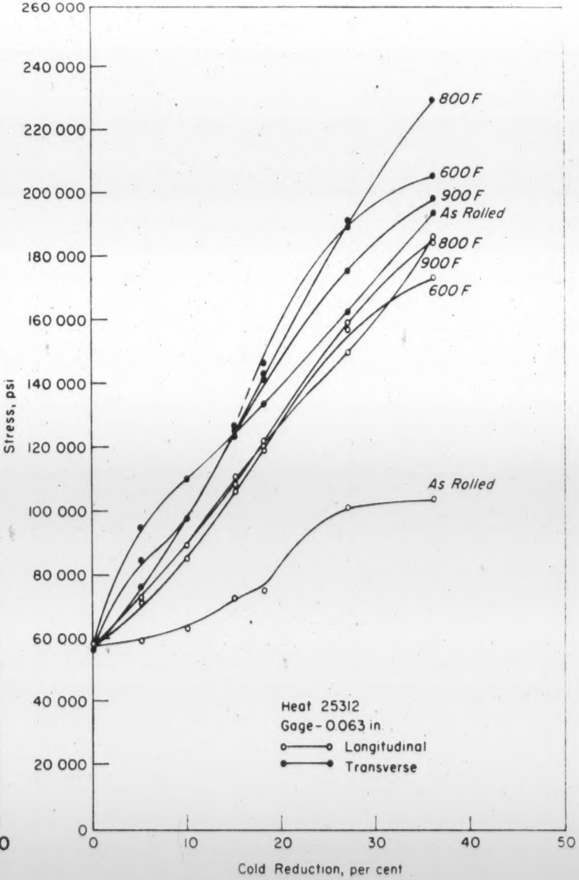

FIG. 10.—The Effects of Cold Reduction and of Stress Relieving at 600 F, 800 F, and 900 F on the Compressive Yield Strength.

TABLE IV.—MECHANICAL PROPERTIES OF HEAT 69346, 0.066 IN. GAGE.

Cold Reduction, per cent	Tension				Compression	
	Yield Strength, 0.2 per cent offset, psi	Tensile Strength, psi	Elongation in 2 in., per cent	Young's Modulus, *E*, psi	Yield Strength 0.2 per cent offset, psi	Young's Modulus, *E*, psi
As Rolled—Longitudinal						
0	49 670	105 535	56.5	29.0×10^6	54 095	30.7×10^6
5	76 589	117 150	42.5	29.2	55 890	29.6
10	90 260	125 790	34.8	30.4	63 910	28.3
15	107 245	139 465	25.8	28.9	72 430	26.8
21	121 745	145 585	23.0	27.8	73 965	27.7
29	150 500	171 500	13.8	25.7	78 995	28.2
40	163 955	189 145	10.0	26.6	86 765	27.3
As Rolled—Transverse						
0	51 250	106 745	55.2	28.5×10^6	53 015	31.9×10^6
5	83 395	115 415	42.0	29.3	90 780	30.0
10	94 765	122 560	33.5	29.9	112 725	27.9
15	112 250	140 240	21.8	29.4	129 300	28.0
21	117 755	147 250	18.8	26.7	137 800	30.2
29	135 415	174 345	11.5	28.0	160 725	30.1
40	158 565	195 645	8.0	27.5	186 350	29.0
Stress Relieved at 600 F—Longitudinal						
5	77 030	115 945	42.8	31.9×10^6	75 770	30.4×10^6
10	93 765	124 375	34.3	29.6	91 625	31.5
15	115 990	141 125	25.8	28.7	103 700	30.6
21	125 860	147 090	23.0	30.0	111 300	28.8
29	158 700	178 140	10.3	28.3	125 975	30.1
40	177 605	202 075	8.3	29.5	146 150	30.3
Stress Relieved at 600 F—Transverse						
5	81 590	115 335	42.5	33.2×10^6	92 910	30.3×10^6
10	95 310	122 770	33.5	30.5	106 750	30.1
15	117 345	140 785	22.8	30.3	132 225	28.9
21	123 420	148 695	20.0	29.8	139 525	30.8
29	148 395	179 530	9.5	29.2	174 425	31.7
40	172 750	204 850	6.0	30.8	217 750	32.5
Stress Relieved at 800 F—Longitudinal						
5	79 645	114 835	43.5	30.5×10^6	73 585	30.7×10^6
10	100 510	128 705	34.0	27.5	90 840	30.4
15	118 465	141 410	26.3	28.4	108 400	30.3
21	130 330	149 275	22.8	27.6	122 925	32.0
29	160 270	174 350	12.0	28.0	140 800	31.1
40	181 860	208 870	8.8	28.5	152 175	30.5
Stress Relieved at 800 F—Transverse						
5	81 570	115 840	40.8	29.9×10^6	86 835	31.1×10^6
10	97 550	121 590	33.0	27.8	106 350	32.8
15	119 645	142 850	22.8	26.7	134 400	29.9
21	125 140	148 140	20.3	27.9	141 700	31.1
29	153 940	179 235	11.3	29.0	168 825	31.6
40	185 100	213 825	6.8	30.9	203 700	33.2
Stress Relieved at 900 F—Longitudinal						
5	78 355	116 895	41.0	29.4×10^6	77 845	30.7×10^6
10	94 260	125 870	34.8	29.3	99 825	29.8
15	116 970	142 795	25.8	28.6	111 775	29.8
21	121 400	145 305	24.8	29.5	127 175	30.6
29	150 895	174 425	14.3	27.6	157 075	31.4
40	179 270	199 250	9.5	26.8	166 675	30.1
Stress Relieved at 900 F—Transverse						
5	81 985	116 235	41.0	29.4×10^6	88 170	32.7×10^6
10	97 600	126 080	32.8	28.5	108 975	33.1
15	117 830	142 050	23.3	27.7	132 425	30.0
21	126 690	150 680	19.5	28.9	147 000	33.4
29	151 275	180 715	11.3	29.7	181 625	30.5
40	178 990	210 460	7.3	30.5	205 650	31.2

TABLE III.—MECHANICAL PROPERTIES OF HEAT 25312, 0.063 IN. GAGE.

Cold Reduction, per cent	Tension				Compression	
	Yield Strength, 0.2 per cent offset, psi	Tensile Strength, psi	Elongation in 2 in., per cent	Young's Modulus, *E*, psi	Yield Strength, 0.2 per cent offset, psi	Young's Modulus, *E*, psi
			As Rolled—Longitudinal			
0	57 030	121 055	54.0	30.1 × 10^6	57 170	29.3 × 10^6
5	72 175	137 050	42.5	28.9	58 660	22.5
10	81 900	142 215	37.3	28.7	62 910	27.7
15	104 915	162 560	28.3	27.1	73 825	25.4
18	114 665	166 975	26.8	27.1	74 250	27.6
27	144 495	192 390	19.5	26.1	101 000	26.8
36	161 110	213 145	17.3	26.8	103 375	27.1
			As Rolled—Transverse			
0	51 575	119 520	55.0	32.3 × 10^6	56 480	32.1 × 10^6
5	83 015	133 510	42.3	29.6	95 270	29.3
10	96 080	145 465	33.0	29.6	109 500	29.9
15	102 040	161 230	25.5	29.7	125 825	29.2
18	110 170	167 275	22.8	28.4	133 200	30.0
27	130 680	196 065	16.3	28.2	162 275	28.9
36	144 575	214 650	13.8	29.4	193 675	30.7
			Stress Relieved at 600 F—Longitudinal			
5	70 100	130 050	46.5	28.6	71 040	26.9 × 10^6
10	87 320	138 225	39.0	27.6	84 215	27.6
15	104 550	149 300	32.0	32.5	106 055	28.3
18	119 250	155 250	28.5	28.5	118 285	27.8
27	162 850	191 125	14.0	29.0	156 110	27.3
36	193 350	215 725	7.0	28.2	173 450	29.5
			Stress Relieved at 600 F—Transverse			
5	73 180	130 500	44.8	28.7 × 10^6	84 210	27.6 × 10^6
10	87 095	135 675	38.8	31.1	101 735	31.4
15	110 175	149 450	29.3	31.2	105 225	27.6
18	121 350	157 350	26.0	30.4	146 720	28.1
27	158 750	191 715	11.3	31.5	189 600	30.6
36	182 500	216 250	9.3	30.7	205 705	30.1
			Stress Relieved at 800 F—Longitudinal			
5	69 115	129 775	47.8	34.0 × 10^6	72 400	30.0 × 10^6
10	83 320	135 700	41.3	33.5	89 175	31.6
15	108 375	156 025	30.8	31.9	107 570	31.6
18	124 050	162 700	28.3	29.2	118 975	30.2
27	155 325	191 600	13.5	29.3	159 420	28.7
36	193 000	229 450	6.8	28.7	184 740	30.0
			Stress Relieved at 800 F—Transverse			
5	68 985	127 725	46.8	33.2 × 10^6	84 275	29.1 × 10^6
10	86 300	135 275	39.5	31.7	96 635	29.3
15	109 250	150 375	30.0	33.2	125 040	27.8
18	119 575	161 425	25.8	34.3	141 370	30.5
27	160 250	196 000	10.3	30.3	191 685	31.6
36	185 550	221 500	7.5	29.4	229 755	31.8
			Stress Relieved at 900 F—Longitudinal			
5	71 670	123 265	45.0	29.1 × 10^6	71 510	28.2 × 10^6
10	85 115	130 100	40.5	28.2	85 520	31.1
15	109 420	153 600	32.3	27.8	110 345	29.7
18	115 805	154 530	31.0	25.9	120 845	28.5
27	145 935	172 825	18.5	25.7	149 735	28.1
36	178 250	202 970	6.8	25.4	186 080	28.4
			Stress Relieved at 900 F—Transverse			
5	74 465	129 110	45.3	28.2	75 940	29.6 × 10^6
10	89 580	133 325	39.8	27.0	97 100	29.6
15	106 785	148 305	32.0	28.4	126 355	30.7
18	120 880	154 650	29.8	25.8	142 470	31.4
27	155 600	184 845	9.5	30.1	175 025	31.8
36	183 110	210 315	5.5	27.1	197 855	32.7

yield strength is lower at low cold reductions but exceeds the transverse yield strength above 15 per cent cold reduction. The data in Tables III and IV show only a very slight difference in elongation. These observations do not hold for compression. Figures 9 and 10 show that the compressive yield strength of cold-rolled material is substantially lower in the longitudinal direction, and that the difference in longitudinal and transverse yield strengths increases with increasing cold reductions. It is also apparent that the longitudinal yield strength is considerably lower in compression than in tension. This characteristic is a well-known phenomenon in type 301 steels.

Heat No. 69346, which has a more stable austenitic microstructure, manifests a very small difference between tensile strength in the longitudinal and transverse directions. This is apparent in Fig. 11 where it will also be obvious that the yield strength shows the same tendency as in heat No. 25312. It is lower in the longitudinal than in the transverse direction at low cold reductions, but eventually the relationship is reversed as cold reduction increases. Again, Table IV reveals essentially no difference in elongation. One can see in Figs. 11 and 12 that the same difference in longitudinal and transverse yield strengths in compression is developed in this heat as well, and that again the

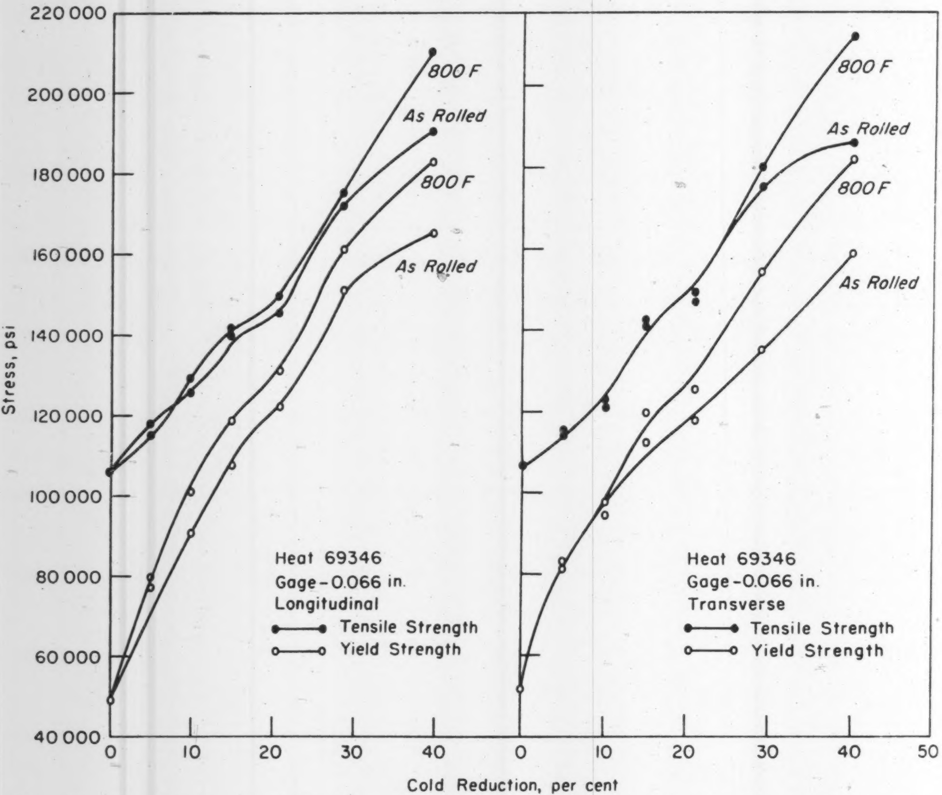

FIG. 11.—The Effects of Cold Reduction and of Stress Relieving at 800 F on Tensile Properties.

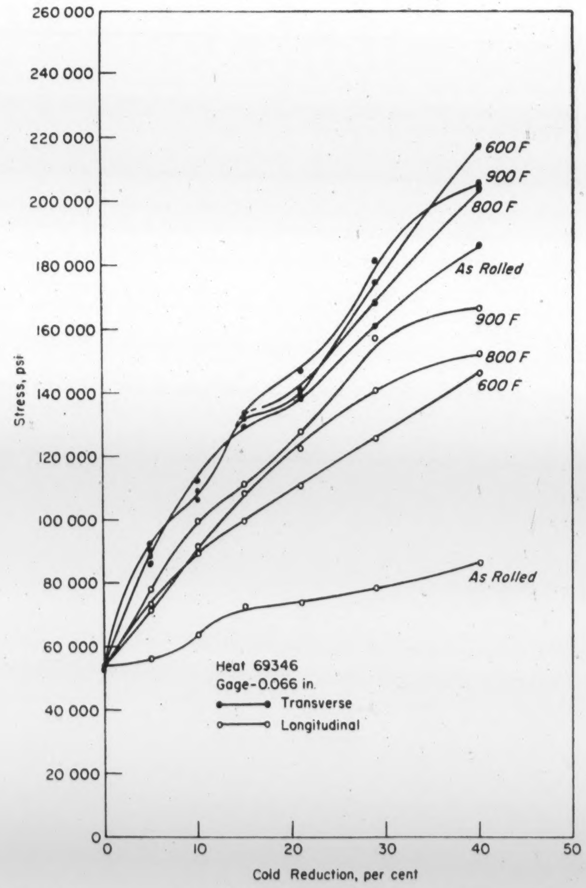

FIG. 12.—The Effects of Cold Reduction and of Stress Relieving at 600 F, 800 F, and 900 F on the Compressive Yield Strength.

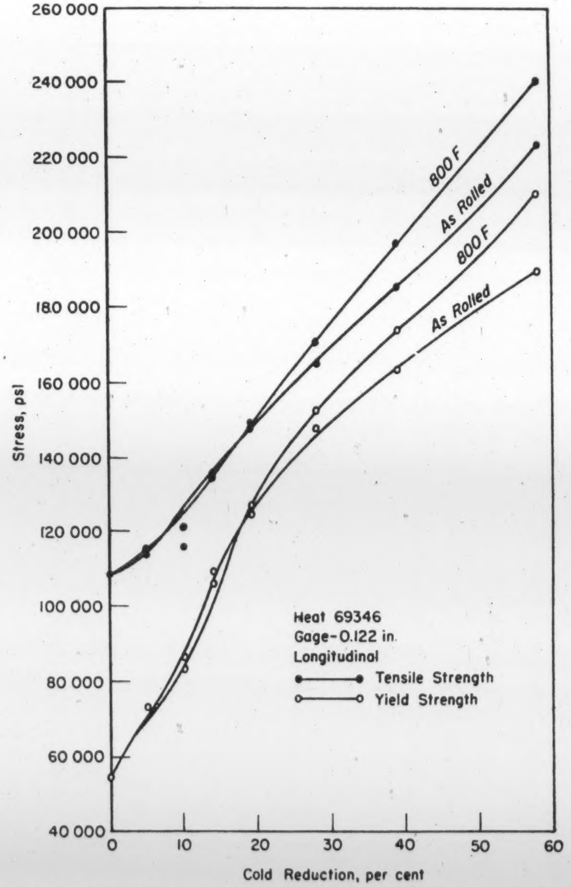

FIG. 13.—The Effects of Cold Reduction and of Stress Relieving at 800 F on Tensile Properties.

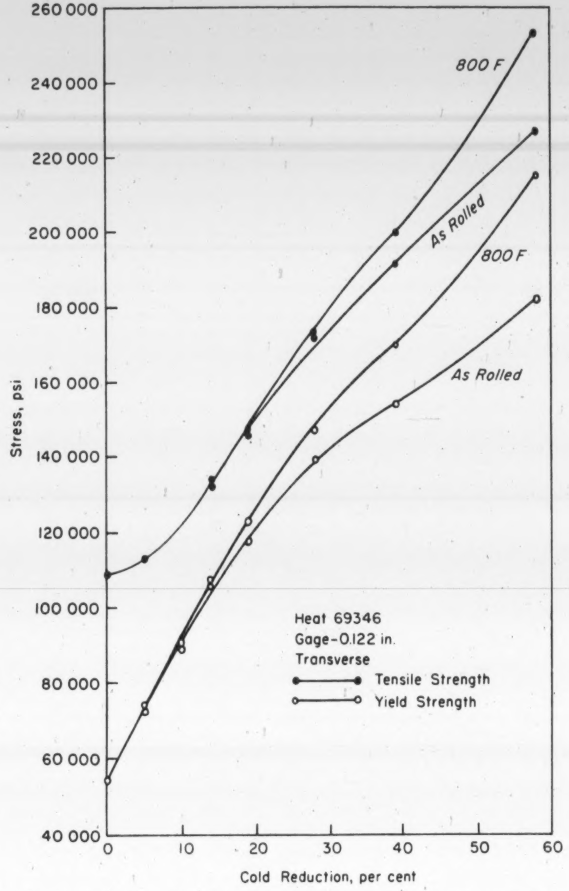

FIG. 14.—The Effects of Cold Reduction and of Stress Relieving at 800 F on Tensile Properties.

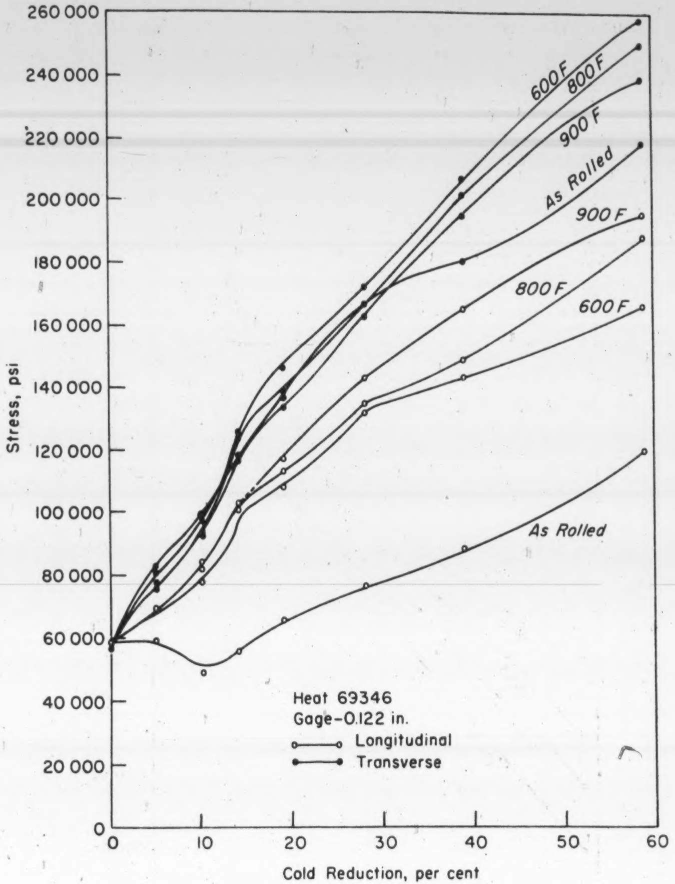

FIG. 15.—The Effects of Cold Reduction and of Stress Relieving at 600 F, 800 F, and 900 F on the Compressive Yield Strength.

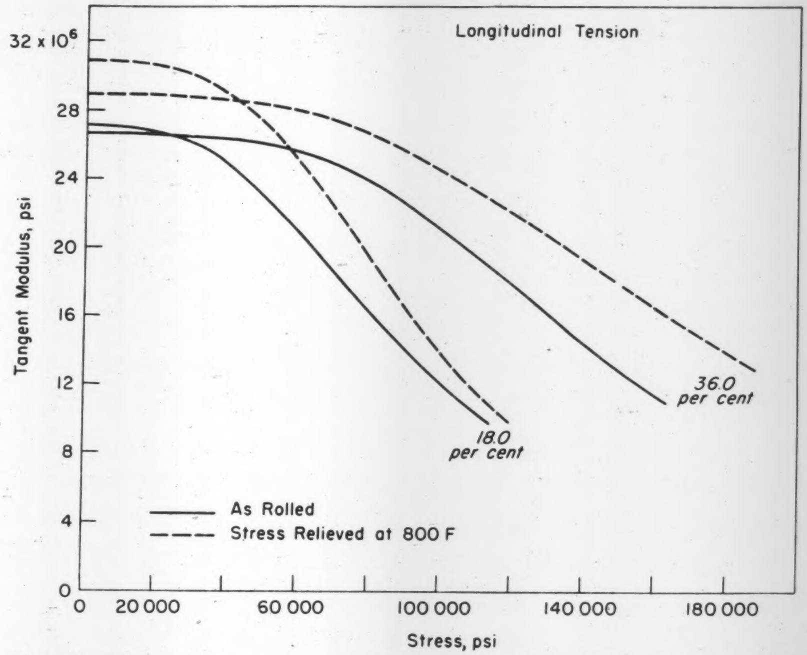

FIG. 16.—The Effects of 18 per cent and 36 per cent Cold Reductions and of Stress Relieving on the Tangent Modulus. Heat 25312.

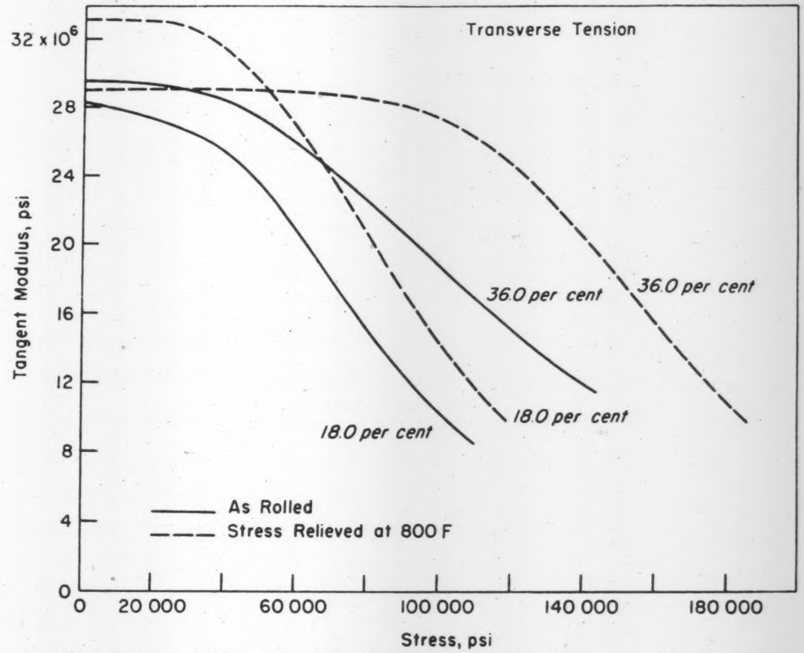

FIG. 17.—The Effects of 18 per cent and 36 per cent Cold Reductions and of Stress Relieving on the Tangent Modulus. Heat 25312.

TABLE V.—MECHANICAL PROPERTIES OF HEAT 69346, 0.122 IN. GAGE.

Cold Reduction, per cent	Tension				Compression	
	Yield Strength, 0.2 per cent offset, psi	Tensile Strength, psi	Elongation in 2 in., per cent	Young's Modulus, *E*, psi	Yield Strength, 0.2 per cent offset, psi	Young's Modulus, *E*, psi
			As Rolled—Longitudinal			
0	54 915	108 675	51.8	30.4 × 10^6	58 510	29.4 × 10^6
5	73 290	114 825	45.0	26.6	59 465	28.2
10	83 655	116 175	39.5	25.6	48 370	27.9
14	106 325	135 925	29.0	26.4	56 160	28.7
19	126 250	147 475	22.5	24.9	65 315	27.8
28	147 700	164 550	16.3	26.3	76 400	28.7
39	163 500	184 950	11.3	26.3	88 050	27.3
58	189 425	222 700	6.5	24.5	119 905	25.7
			As Rolled—Transverse			
0	54 610	108 575	51.0	28.4 × 10^6	57 465	29.9 × 10^6
5	74 550	111 600	46.3	28.4	83 470	28.2
10	89 645	119 850	37.0	27.6	99 800	28.5
14	106 425	134 350	26.0	26.5	118 490	27.5
19	117 900	145 900	18.3	26.1	137 435	28.2
28	139 800	171 800	13.5	27.1	167 545	30.5
39	154 275	191 400	10.0	27.2	180 950	28.1
58	182 925	227 775	6.3	31.8	218 850	30.8
			Stress Relieved at 600 F—Longitudinal			
5	75 740	114 950	44.8	28.8 × 10^6	69 560	29.6 × 10^6
10	87 100	121 175	40.0	28.5	77 020	30.5
14	110 800	134 475	30.3	28.3	100 130	32.0
19	128 050	148 850	22.5	29.7	108 675	28.6
28	152 850	174 375	15.3	28.6	132 835	28.7
39	173 850	196 750	8.8	29.0	144 225	32.3
58	205 900	234 175	4.0	29.5	166 375	29.9
			Stress Relieved at 600 F—Transverse			
5	72 230	110 425	47.0	30.5 × 10^6	81 555	31.8 × 10^6
10	89 560	119 550	37.5	28.6	96 905	29.5
14	109 380	132 820	28.8	25.9	125 930	29.2
19	124 925	149 650	20.0	30.3	147 120	31.1
28	150 610	176 270	11.5	26.8	172 535	29.8
39	176 575	200 480	8.3	28.3	207 680	29.5
58	208 775	248 050	4.3	30.3	258 655	31.2
			Stress Relieved at 800 F—Longitudinal			
5	73 760	115 200	45.0	29.4 × 10^6	70 010	29.1 × 10^6
10	87 020	121 125	39.0	28.6	82 330	28.0
14	109 525	135 250	27.5	28.7	102 750	27.1
19	125 700	148 450	23.0	29.7	113 475	28.4
28	153 025	170 500	15.8	28.6	134 985	29.1
39	173 350	196 250	10.5	27.4	149 645	28.5
58	209 450	240 185	5.0	27.0	188 570	27.1
			Stress Relieved at 800 F—Transverse			
5	72 810	112 050	45.5	28.4 × 10^6	77 490	29.4 × 10^6
10	90 085	119 650	37.5	28.7	93 390	28.3
14	107 375	132 775	23.0	27.7	123 380	28.8
19	123 325	147 800	21.0	28.3	138 210	29.4
28	147 800	173 575	11.8	27.1	167 595	30.7
39	170 500	200 850	8.3	29.8	200 265	29.9
58	209 580	254 735	4.8	30.6	250 380	32.7

TABLE V.—*Concluded.*

Cold Reduction, per cent	Tension				Compression	
	Yield Strength, 0.2 per cent offset, psi	Tensile Strength, psi	Elongation in 2 in., per cent	Young's Modulus, E, psi	Yield Strength, 0.2 per cent offset, psi	Young's Modulus, E, psi
STRESS RELIEVED AT 900 F—LONGITUDINAL						
5	71 945	113 860	45.3	34.9×10^6	69 870	30.2×10^6
10	84 240	121 950	41.8	35.3	83 580	30.6
14	105 365	135 550	30.3	30.6	100 680	31.7
19	124 305	149 240	25.5	32.2	117 640	31.1
28	149 530	171 010	17.3	29.2	143 485	30.9
39	171 435	195 230	12.0	29.3	165 265	29.8
58	198 195	231 565	4.3	28.6	195 810	26.5
STRESS RELIEVED AT 900 F—TRANSVERSE						
5	67 940	109 815	47.3	38.6×10^6	76 845	30.9×10^6
10	87 065	120 435	38.0	33.5	94 755	31.1
14	110 940	136 135	27.5	32.3	117 685	30.3
19	123 830	149 190	22.3	32.6	133 480	31.2
28	148 490	168 635	15.0	30.5	163 850	30.8
39	171 475	198 350	9.5	29.3	195 205	30.8
58	207 950	246 305	5.5	31.8	239 750	31.1

longitudinal yield strength is lower for compression than for tension.

Heat No. 69346 was tested in two thicknesses. In the cold-rolled state, the thicker gage has slightly lower strength and higher elongation (Figs. 13 to 15, Table V).

Influence of Austenite Stability:

It has been mentioned that the two heats used in this investigation were deliberately selected because they differed in the degree of austenite stability. Heat No. 25312, the less stable, has substantially higher tensile strength and elongation than heat No. 69346 and in both directions. By comparison of Figs. 9 and 11 one sees, however, that the yield strengths differ only slightly. Figures 10 and 12, when compared, show that in compression, heat 25312 does have higher yield strength in both directions.

Stress Relieving:

The importance of the stress-relieving treatment for cold-rolled sheet lies in its ability to alleviate anisotropy of the mechanical properties. The stress-relieving treatments in this investigation were made at 600, 800, and 900 F for 12 hr. The results of the tests are given in Tables III, IV, and V and are plotted in Figs. 9 to 15.

The general effect of stress relieving is to increase the tensile properties, but a certain amount of cold reduction is necessary before the increase becomes evident. The influence of temperature is not consistent, and in Figs. 9 and 11 only the 800 F treatment is plotted. Figures 10 and 12 indicate that in compression, the transverse yield strength is increased by stress relieving and as expected, the most important result of such treatments is the increase in longitudinal yield strength. Temperature plays a more important role with these steels as indicated by the increased effectiveness, in general, of 800 F and 900 F treatments compared to 600 F. As shown in Figs. 10 and 12, after stress relieving, the compressive yield strength in the longitudinal direction approaches but, at high cold reduction, does not quite attain the same levels as the transverse yield strength.

cold rolling. In the stress-relieved condition then, the modulus at zero stress is approximately constant.

The calculated values of tangent modulus have been plotted in Figs. 16 through 19 for heat No. 25312 for 18 per cent and 36 per cent cold reductions. Both longitudinal and transverse values are given for tension and compression. The tangent modulus in the longitudinal direction is much lower in compression than in tension. This difference is completely eliminated by the stress-relieving treatment. Conversely, in the transverse direction, values of the tangent modulus in tension are lower than the corresponding compressive values. Again, the stress-relieving treatment raises the value of the tangent modulus.

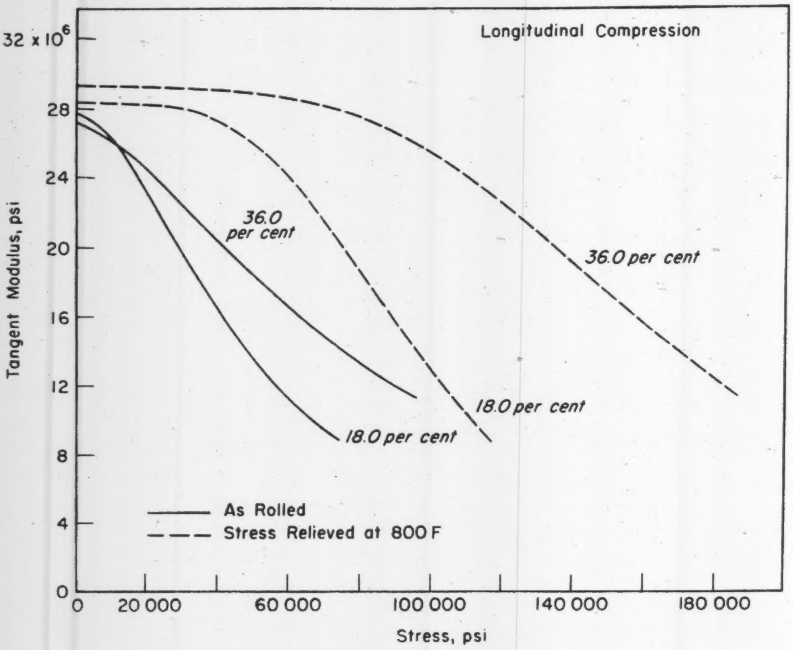

FIG. 18.—The Effects of 18 per cent and 36 per cent Cold Reductions and of Stress Relieving on the Tangent Modulus. Heat 25312.

Modulus:

The experimental values of Young's modulus are recorded in Tables III, IV, and V and show that in both tension and compression the longitudinal modulus decreases with increasing cold reduction. In the transverse direction, the modulus follows no consistent trend, remaining essentially constant.

The stress-relieving treatment at all three temperatures tends to nullify the decrease in initial modulus produced by

Combined Moduli for Use in Compression:

The buckling loads of slender columns in the elastic range can be calculated by Euler's formula. Von Karman proved by very accurate column tests that the Euler formula can also be applied to the plastic range if the initial modulus, E_o, is replaced by the reduced modulus.

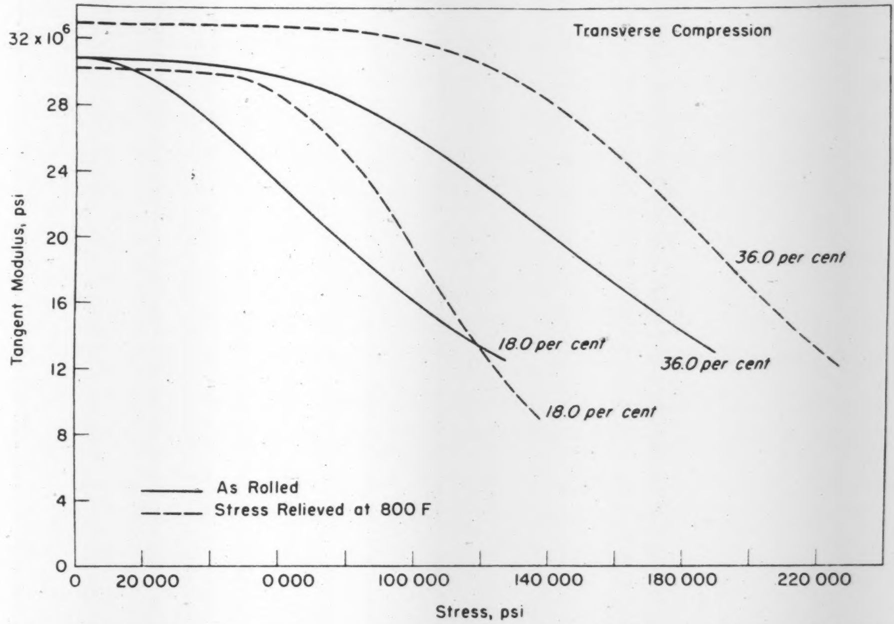

FIG. 19.—The Effects of 18 per cent and 36 per cent Cold Reductions and of Stress Relieving on the Tangent Modulus. Heat 25312.

Timoshenko,[4] in his treatment of centrally-compressed bars in ranges beyond the limit of proportionality also used a reduced modulus which combined the differences in the slopes of the stress-strain curves during loading and unloading.

This concept has been adapted by Watter and Lincoln (3) to the description of the properties of high-strength cold-rolled stainless steel. Their expression of reduced modulus, taking into consideration the directional properties of cold-rolled sheet, is written as follows:

$$E_{rLC} = \frac{4E_{oLC}E_{tLC}}{(E_{tLC}{}^{1/2} + E_{oLC}{}^{1/2})^2}$$

where:

E_{rLC} = longitudinal reduced compression modulus

E_{oLC} = initial modulus in longitudinal compression at zero stress, and

E_{tLC} = tangent modulus in longitudinal compression at a given stress,

$$E_{rTC} = \frac{4E_{oTC}E_{tTC}}{(E_{oTC}{}^{1/2} + E_{tTC})^2}$$

where:

E_{rTC} = transverse reduced compression modulus,

E_{oTC} = initial modulus in transverse compression at zero stress, and

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[4] Timoshenko, "Theory of Elastic Stability," McGraw-Hill Book Co., p. 156 (1936).

E_{tTC} = tangent modulus in transverse compression at a given stress.

The reduced modulus has been calculated for heat No. 25312 for 18 per cent and 36 per cent cold reductions. Figure 20 shows the longitudinal reduced compression modulus as a function of stress for cold-rolled and for stress-relieved material. Figure 21 is a similar plot for the transverse reduced compression modulus.

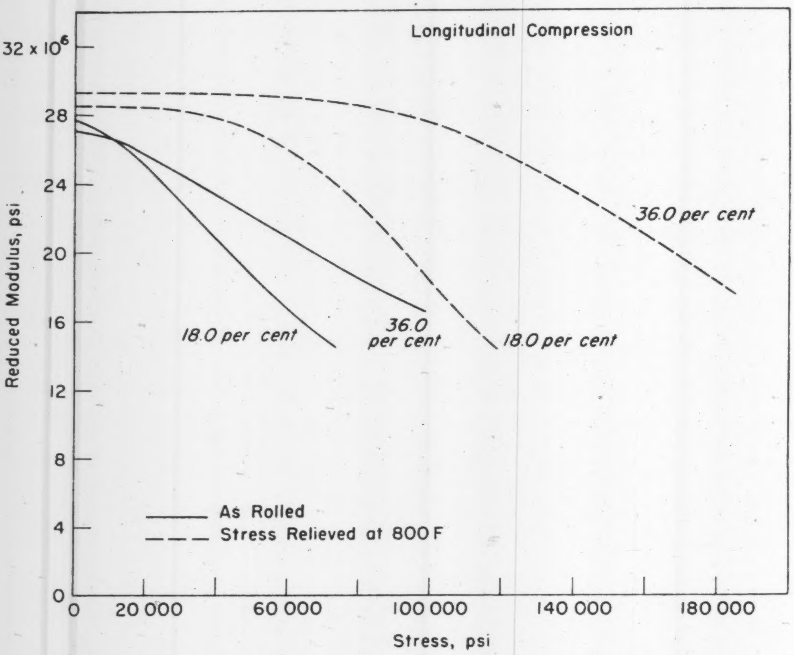

FIG. 20.—Combined Modulus for As-Rolled and for Stress-Relieved Material for 18 per cent and 36 per cent Cold Reductions. Heat 25312.

SUMMARY

The tensile and compressive mechanical properties of two commercial type 201 heats have been determined for both the annealed and the cold-rolled conditions.

In the annealed state, the tensile properties in the longitudinal and transverse directions are approximately the same and the corresponding stress-strain curves superpose. This holds for compressive tests as well, but the compressive stress-strain curves, consequently yield strengths, are always higher than the corresponding tensile properties.

For cold-reduced sheet, the tensile properties in the longitudinal and transverse directions show only slight differences. In compression, the yield strength is substantially lower in the longitudinal direction. This is a familiar phenomenon in type 301 steels.

Of the two heats studied, the less stable has higher tensile strength and undergoes greater elongation before failure. The compressive yield strength is also higher but the yield strength in tension is nearly the same for both heats.

For equivalent amounts of cold reduction, there are no substantial differences in properties for the two gages investigated.

The general effect of stress relieving is to minimize the differences between longitudinal and transverse properties of cold reduced sheet. The effect becomes more pronounced with increasing amounts of cold reduction. The most important specific effect of stress relieving is to increase the modulus and the compressive yield strength in the longitudinal direction. Stress relieving at 800 F or 900 F may have some slight advantage over 600 F treatments.

The modulus in the transverse direction remains approximately constant while the initial longitudinal modulus in tension and compression is reduced by cold rolling.

This decrease in modulus is nullified by stress relieving.

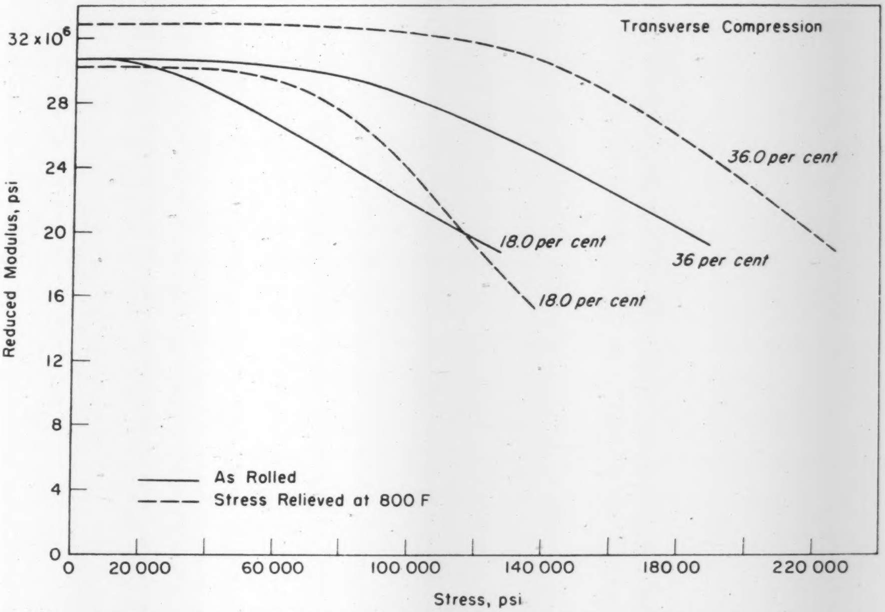

FIG. 21.—Combined Modulus for As-Rolled and for Stress-Relieved Material for 18 per cent and 36 per cent Cold Reductions. Heat 25312.

The compressive tangent modulus of 18 and 36 per cent cold-reduced sheet is lower than the tensile modulus in the longitudinal direction. The relationship is reversed in the transverse direction. These differences are somewhat alleviated by stress relieving.

The reduced modulus has also been calculated and plotted for 18 and 36 per cent cold reductions.

Acknowledgment:

The authors wish to express their appreciation to R. Dreher, R. Hall, and B. Clover who carried out all the tension and compressive tests and to W. Curry and J. Kisiel who performed the metallographic and photographic work.

DISCUSSION

Mr. J. O. Turner.[1]—At our laboratory in Berkeley and in Livermore, there are a number of large nuclear particle accelerators which depend upon very, very high magnetic fields, of the order of 23 kilogauss and up, and structures must be built to operate in these magnetic fields. These structures have to be of lowest magnetic permeability and also have high mechanical strength. Therefore, chromium-nickel stainless steels with as low magnetic permeability as possible have been used. We should be interested in knowing if any other investigators in this field have developed any information on the magnetic properties of these new alloys as affected by welding and cold working and also extremely low temperatures.

Mr. R. A. Walsh (*author*).—Permeability tests have been made on cold-rolled sheet. If it would be of any value, information can be supplied as data for permeability for reductions up to 50 per cent on type 201 sheet. In general the permeability for annealed 201 sheet ought to be about 1.02 maximum.

In contrast to this, a typical value after 20 per cent cold reduction would be about 2.7 and after 30 per cent cold reduction about 7.0. These values would be for μ at 200H.

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[1] University of California, Radiation Laboratory, Berkeley, Calif.

ELECTRON MICROSCOPIC IDENTIFICATION OF THE γ' PHASE OF NICKEL-BASE ALLOYS[1]

By W. C. Bigelow,[2] J. A. Amy,[3] and L. O. Brockway[4]

Synopsis

Electrolytic etching for short periods in a reagent consisting of 5 ml hydrofluoric acid (48 per cent), 10 ml glycerol, 10 to 50 ml ethyl alcohol, and water to make 100 ml total volume, produces a preferential attack on the particles of the intermetallic γ' phase of nickel-base alloys containing titanium and aluminum. A procedure using this reagent has been developed which permits the γ' particles to be distinguished by electron microscopy from particles of carbide and nitride phases which also precipitate in these alloys. The use of this procedure in studies of the distribution of the γ' phase in several commercial alloys is described.

Numerous investigations have shown that the high resolutions and magnifications attainable by electron microscopy provide many advantages in studying the microstructures of metals and alloys, particularly in determining the size, shape, and distribution of minor-phase particles on a submicroscopic scale. The usefulness of electron metallographic techniques could be considerably extended, however, if selective etching procedures could be developed which would permit different minor phases to be identified or distinguished by their appearance in the electron micrographs. Such procedures have been widely used in optical metallography, but have not been adapted to electron metallography because of the basic differences in the two techniques. The optical etchants usually function by staining particles of different phases different colors which are readily observed when the etched surfaces are examined in the light microscope. In electron microscopy, the illuminating radiation is not sensitive to color differences in the usual sense; furthermore, the surfaces are normally examined indirectly through the use of surface replicas, so that the stained particles are not a part of the specimen actually examined.

These factors must be taken into consideration in developing etching procedures for distinguishing different minor phases in electron microscopy. The two most promising approaches are: (1) the use of reagents leaving loosely adherent chemical products on the minor-phase particles which could be picked up by surface replicas and used as a basis for distinguishing or identifying them; (2) the use of reagents which preferentially attack certain minor phases so that the resulting differences in the shape or relief of the particles could be readily detected in the electron microscope through the use of shadowing techniques.

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[1] This research was carried out through the Engineering Research Institute of the University of Michigan under contract AF-33(616)-23 for the Metallurgy Branch of the Aeronautical Research Laboratory (WCRRL), Wright Air Development Center, Wright-Patterson Air Force Base, Ohio.

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[3] Research Assistant, Engineering Research Institute, University of Michigan.

[4] Professor of Chemistry, Department of Chemistry, University of Michigan.

In recent studies of the nickel-base alloys containing titanium and aluminum, an etching procedure of this latter type has been developed which permits the intermetallic phase of these alloys to be distinguished from the carbide and nitride precipitates. This phase is generally considered to be the γ' phase which has a structure based on that of Ni_3Al phase of the nickel-aluminum system. It therefore differs considerably in its susceptibility to chemical attack from the carbide and nitride phases. Advantage is taken of this fact and a reagent is used which preferentially dissolves the γ' particles but which does not attack the carbide and nitride particles. Surfaces treated with this reagent show depressions at the locations of the γ' particles while the carbide and nitride particles remain in relief.

TABLE I.—COMPOSITION OF ETCHING REAGENTS.

Reagent	Composition
A........	5 ml hydrofluoric acid (48 per cent), 10 ml glycerol, 85 ml ethyl alcohol
B........	12 ml phosphoric acid (85 per cent), 47 ml sulfuric acid (96 per cent), 41 ml nitric acid (70 per cent)
C........	5 ml hydrofluoric acid (48 per cent), 10 ml glycerol, 10 to 50 ml ethyl alcohol, water to make 100 ml total volume

PROCEDURE

The procedure usually, though not necessarily, involves a preliminary etching of the specimens with reagents which attack the matrix phase preferentially and leave the particles of the γ' phase and of the carbide and nitride phases protruding from the etched surfaces. Reagents *A* and *B* of Table I have been found to be useful for this purpose. The selective attack on the γ' particles is produced by subsequent etching of these surfaces with reagent *C*. Comparison of micrographs after this etch with those taken after the preliminary etch permits the size, shape, and distribution of the γ' particles to be determined.

The etching treatments with all these reagents are carried out electrolytically. For unmounted specimens about 1 by ½ by ½ in. in size, currents of about 0.2 amp are used. Etching in reagents *A* and *B* usually requires from 3 to 10 sec. The period of etching in reagent *C* is usually less than 1 sec and care is required to avoid over-etching. Equally good results have been obtained with specimens polished mechanically and electrolytically prior to etching. When mechanical polishing is used, particular care must be exercised to avoid disturbing the structure of the surfaces by cold work. For electrolytic polishing, very good results have been obtained with several different nickel-base alloys by using a polishing solution consisting of 90 parts glacial acetic acid to 10 parts perchloric acid (70 to 72 per cent).

Reagents *A* and *C* are both modifications of the reagent of Taylor and Floyd (1)[5] (10 per cent glycerol, 5 per cent hydrofluoric acid, 85 per cent water) in which all or part of the water is replaced by ethyl alcohol. The quantity of alcohol added has considerable influence on the etching characteristics of the reagent, however. Taylor and Floyd's reagent produces a preferential attack on the γ' particles similar to

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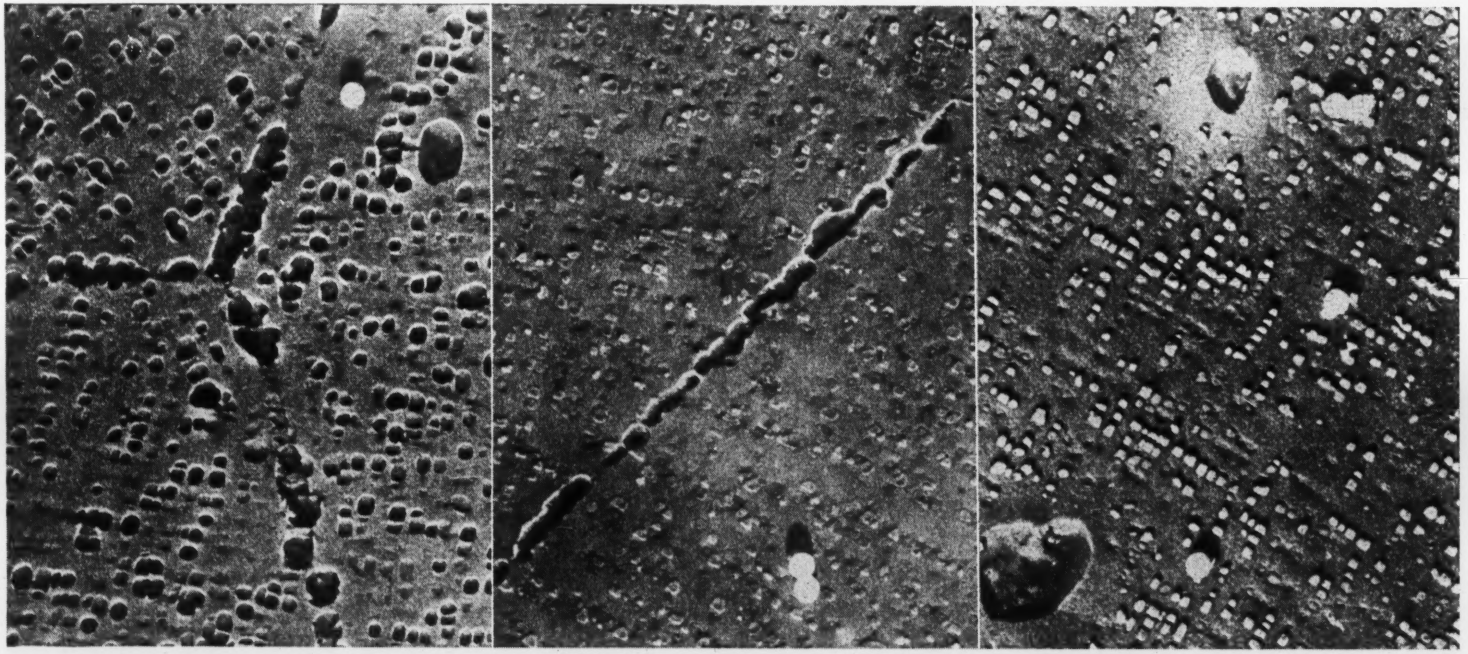
[5] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 952.



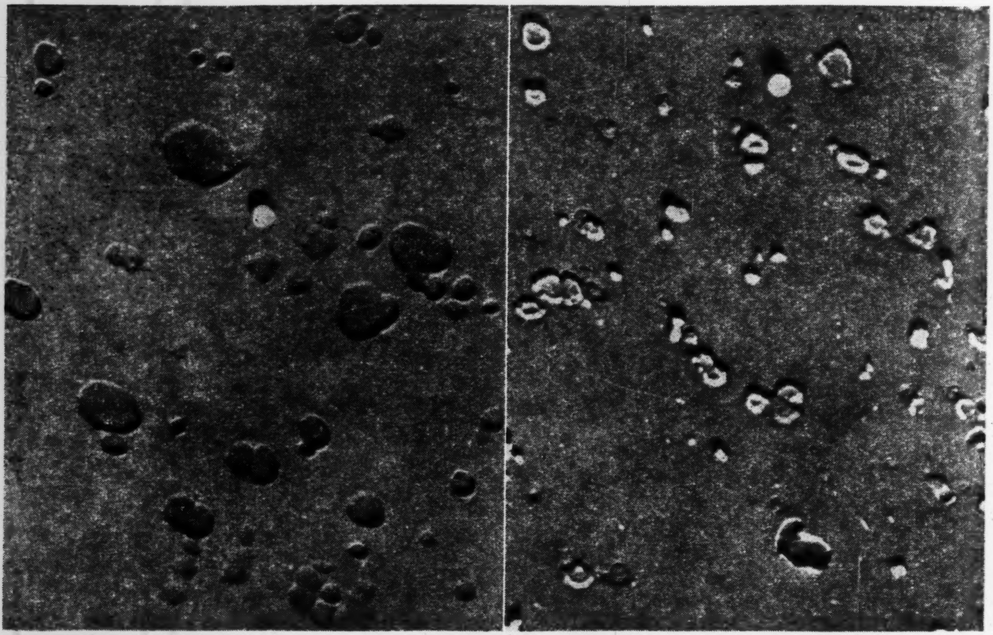
(*a*) Preliminary etch with reagent *A*. All minor phase particles in relief.

(*b*) Etched ½ sec in reagent *C*. Grain boundary particles in relief, γ' particles level with surface.

(*c*) Etched 1 sec in reagent *C*. Large carbide particles in relief, γ' particles etched out.

FIG. 1.—Electron Micrographs from a Specimen of Inconel-X Alloy Aged 1000 hr at 1400 F, Showing Results of Selective Etching Procedure (× 12,000).

reagent *C*, but in many cases it also attacks the grain boundaries and the interfaces around the carbide and nitride particles rather severely. The addition of small quantities of alcohol, as in reagent *C*, reduces this interfacial attack, permitting the γ' particles to be more readily distinguished from the carbide and nitride particles. When all the water is replaced by alcohol, the action of the reagent is virtually reversed and it attacks the matrix phase more rapidly than the γ' phase. The action of this reagent thus can be varied over wide ranges by altering its composition. Reagent *B* is frequently used in electrolytic polishing of stainless steels (2), but is also an excellent etchant for preparing many heat-resistant alloys for electron microscopic studies when used with low etching currents.



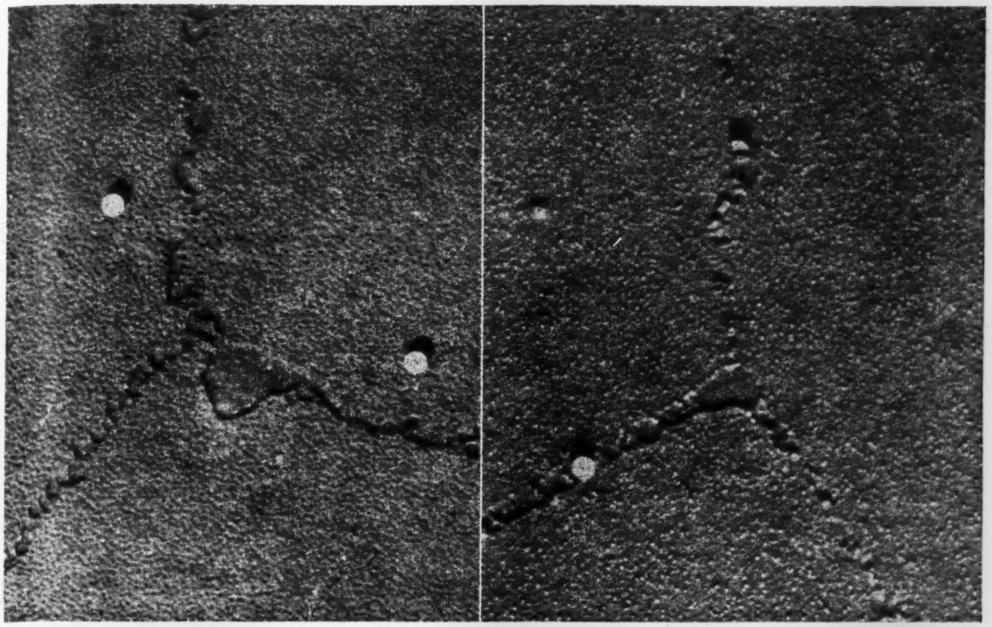
(*a*) Preliminary etch with reagent *A*. All minor phase particles in relief.

(*b*) Final etch with reagent *C*. Carbide particle in relief, γ' particles etched out.

FIG. 2.—Electron Micrographs Showing Selective Etching Attack on a Specimen of Inconel-X Alloy Aged 1000 hr at 1600 F (× 12,000).

RESULTS

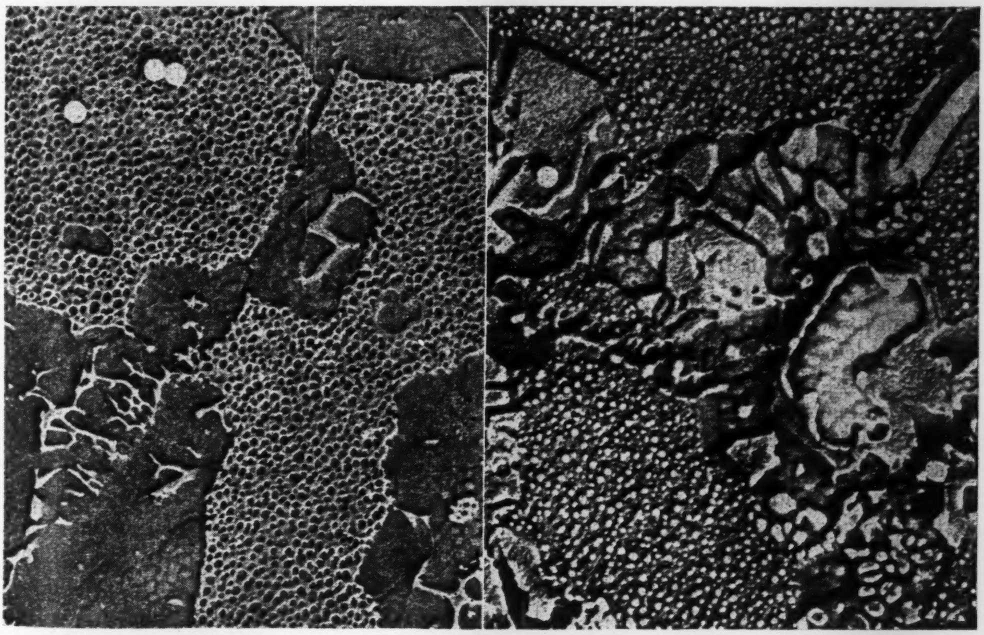
Some typical results which have been obtained with this etching procedure in studies of the microstructures of the commercial nickel-base alloys, Inconel-X, Waspalloy, and Udimet, are illustrated by the electron micrographs of Figs. 1 to 4. Typical compositions for these alloys are given in Table II. Micrograph (*a*) of each figure shows the microstructure of the alloy specimens as revealed by the preliminary etching treatment, while micrographs (*b*) and (*c*) show the same surfaces after subsequent etching with reagent *C* to dissolve the γ' particles. These micrographs were obtained from palladium-shadowed collodion replicas of the etched surfaces and were photographically processed for reproduction here so that the shadows ap-



(*a*) Preliminary etch with reagent *B*. All minor phases in relief.

(*b*) Final etch with reagent *C*. Grain boundary precipitate in relief, γ' particles etched out.

FIG. 3.—Electron Micrographs Showing the Application of the Selective Etching Procedure to a Specimen of Waspalloy Aged 100 hr at 1400 F (× 12,000).



(*a*) Preliminary etch with reagent *B*. All minor phases in relief.

(*b*) Final etch with reagent *C*. γ' Phase etched out, carbide phase at grain boundary in relief.

FIG. 4.—Electron Micrographs Showing the Application of the Selective Etching Procedure to a Specimen of Udimet Alloy Aged 24 hr at 1550 F and 16 hr at 1400 F (× 12,000).

pear black as on the original photographic plates. When interpreting these micrographs in terms of the relief of the minor-phase particles, it is important to remember that the shadowed surfaces of the collodion replicas are negative reproductions of the surfaces of the metal specimens. Thus, particles which protrude from the metal surfaces are reproduced as indentations in the collodion, while indentations in the metal surfaces are reproduced as protrusions from the surfaces of the replicas. The relief of features in the micrographs can be determined from the shadows associated with them. To aid in determining the angle and direction of shadowing, polystyrene latex spheres were placed on the surfaces of the replicas just prior to shadow casting. One or more of these spheres are included in the field of each micrograph. Since these spheres have uniform diameters of 2580 ± 30Å, they also provide a very convenient standard of size (**3**, **4**).

TABLE II.—COMPOSITIONS OF ALLOYS, PER CENT BY WEIGHT.

Alloy	Ni	Cr	Co	Fe	Mo	Ti	Al	Cb	Mn	Si	C
Inconel-X.......	73.4	14.6	...	6.90	...	2.33	0.67	1.04	0.51	0.39	0.05
Waspalloy......	56.5	19.5	13.4	2.90	2.81	2.21	1.09	...	0.88	0.62	0.09
Udimet.........	55.4	19.5	14.8	...	3.80	2.79	2.94	...	0.17	0.50	0.08

The micrographs of Fig. 1 were taken from a specimen of Inconel-X alloy aged 1000 hr at 1400 F. Since the selective etching procedure was, to a considerable extent, developed in studies of the minor phases and microstructure of this particular specimen, some of the evidence that it is selective for the γ' phase will be presented in discussing these micrographs. As shown in micrograph (*a*), there is considerable precipitation both at the grain boundaries and within the matrix grains of this specimen. The particles of the matrix precipitate in this specimen are particularly interesting because of their marked cubic shape and their definite orientation and alignment along preferred directions in the grains. Micrographs (*b*) and (*c*) show this same surface after two subsequent etching treatments of about $\frac{1}{2}$ sec each in reagent *C*. The first of these treatments reduced the cubic particles to nearly the level of the matrix metal, while the second removed them completely, leaving cubic depressions in the surface at the positions they occupied. In neither case is there noticeable attack on the precipitates at the grain boundaries nor on the larger spheroidal particles in the grains. These micrographs clearly show that reagent *C* produces a direct, selective electrochemical attack on the cubic matrix particles and that these particles differ from the other precipitates of the specimen.

The identification of the cubic particles as the intermetallic γ' phase was accomplished by back-reflection X-ray diffraction patterns obtained from surfaces etched as in micrograph (*a*). These patterns showed weak reflections of the γ' phase in close proximity to the reflections from the matrix phase as reported by Nordheim and Grant (**5**, **6**). Since the cubic particles are the only ones present in sufficient numbers to contribute to X-ray diffraction patterns under the conditions used, they must be the γ' phase. In addition, electron diffraction patterns obtained from the surface of the specimen by reflection techniques after etching with reagent *C* showed diffraction rings of an $M_{23}C_6$ type complex carbide, columbium carbonitride, and titanium nitride which were comparable in intensity to those obtained prior to etching, indicating that these carbide and nitride phases were not removed by etching with this reagent. (Direct electron diffraction evidence on the γ' phase could not be obtained, since its structure is so similar to that of the matrix phase that the two cannot be distinguished by this method. Lines which could be attributed to either the γ' or the matrix phase were present in the patterns before and after etching with reagent *C*.) Similar results have been obtained from other alloys and from other specimens of Inconel-X, aged at different temperatures but containing sufficient quantities of the γ' phase to be detected by X-ray diffraction methods.

The micrographs of Fig. 2 were taken from a specimen of Inconel-X alloy aged for 1000 hr at 1600 F. X-ray diffraction patterns from this specimen gave no evidence of the presence of the γ' phase, and micrographs from the specimen etched in the usual manner showed only spheroidal particles (micrograph (*a*)) having the appearance of carbide and nitride particles as observed in micrographs from other alloys. Micrograph (*b*) shows that the majority of these particles are susceptible to attack by reagent *C*, however, indicating that they are not carbide or nitride phases and very probably are the intermetallic γ' phase. The fact that they are spheroidal in form rather than cubic, as in the specimen described above, may well be attributed to different aging of the two specimens. Similar studies on series of specimens aged for different periods at 1200, 1400, and 1600 F provided evidence that the γ' phase precipitates in this alloy under a wide range of aging conditions with a wide variation in the size, shape, and abundance of the γ' particles. In all cases, the γ' particles were found primarily within the matrix grains. A few isolated particles of the carbide and nitride phases were also found in the matrix grains, but for the most part these phases precipitated at the grain boundaries. Electron diffraction studies have identified these phases as including titanium nitride, columbium carbonitride, and an $M_{23}C_6$-type complex carbide (7).

The electron micrographs of Figs. 3 and 4 illustrate the applications of the selective etching procedure to two other nickel-base alloys, Waspalloy and Udimet. Micrograph 3(*a*) shows the microstructure of a specimen of Waspalloy alloy aged 100 hr at 1400 F. Micrograph 4(*a*) shows the microstructure of an Udimet specimen, aged 24 hr at 1550 F and 16 hr at 1400 F. Both these specimens were etched with reagent *B*. Heavy precipitation is evident, both within the matrix grains and at the grain boundaries of both these specimens. Subsequent etching with reagent *C* produced the characteristic attack on the small particles within the matrix grains of both specimens, indicating that these are particles of the intermetallic γ' phase. In the Waspalloy specimen the grain-boundary precipitate was unaffected by reagent *C* and appears to be almost entirely carbide and nitride phases. In the Udimet specimen, however, it appears that considerable amounts of the γ' phase precipitated at the grain boundaries together with the carbide and nitride phases. In both alloys a few isolated carbide and nitride particles are also observed within the matrix grains.

DISCUSSION

Several recent metallurgical investigations (1, 5, 8, 9) have provided evidence that the age-hardening phenomena of the nickel-base alloys containing titanium and aluminum result from the precipitation of the intermetallic γ'

phase. Because of the present technological importance of these alloys, the study of the precipitation of this phase is a matter of practical as well as theoretical importance. Such studies are often very difficult to carry out by the usual methods of physical metallurgy, however. Since the γ' phase has a cubic structure which differs very little in lattice dimensions from that of the matrix phase, it usually cannot be identified by electron diffraction methods and difficulties may arise in X-ray diffraction studies. Optical microscopy is also ineffective in many cases, for, as shown here, the γ' particles seldom become sufficiently large to be resolved by this method. Difficulties may even be encountered in recognizing the γ' phase in conventional electron microscopic studies, due to the variations in the form of the γ' particles in different alloys and with different aging conditions. It therefore appears that electron microscopic studies employing the etching procedures described here may provide one of the most sensitive methods presently available for studying the γ' phase in these alloys.

Finally, it is of interest to note certain structural characteristics which appear to prevail in the nickel base alloys containing titanium and aluminum, as revealed by the electron microscopic studies described here. The intermetallic γ' phase has been observed in aged specimens of all the commercial alloys of this type studied to date. While wide variations occur in their size, shape, and distribution, the γ' particles appear to form predominantly within the matrix grains. Precipitation of appreciable amounts of this phase at the grain boundaries has been observed only in specimens of the Udimet alloy and is thought to be related to the higher titanium and aluminum content of this alloy. Titanium nitride particles and, in the case of alloys containing columbium, columbium carbonitride particles are present in these alloys even after solution treatment at 1975 to 2050 F and water quenching. These particles are randomly distributed and increase in size with subsequent aging treatments. Grain-boundary precipitates are not present in the solution-treated, water-quenched specimens, but develop on aging. These precipitates appear to consist predominantly of complex carbides of the $M_{23}C_6$ type but may include some of the titanium nitride and columbium carbonitride phases.

Acknowledgments:

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THE EFFECT OF SIZE, SHAPE, AND GRAIN SIZE ON THE FATIGUE STRENGTH OF MEDIUM CARBON STEEL*

By Ch. Massonnet[1]

In the last few years, several papers have been published (**1,2,3,4,5,6**)[2] dealing with the design of machine parts based on the knowledge of the fatigue strength of notched parts. Some features of this method of design, such as the effect of the average stress and of the nature of the stress on the resistance to fatigue, are relatively well known.

But our information is less satisfactory concerning the effect of stress variation and notch effect on fatigue strength. The study of the notch effect cannot be dissociated from the study of the size effect, which consists of the decrease (mostly observed in rotative flexure) in endurance limit when the diameter of the specimen increases. The object of this paper is to present experimental data about the size and notch effects, and thereby contribute to the improvement of the design of machine parts.

General Outline of the Tests

While the literature on the size and notch effects is extensive, it is hardly possible to draw from it any precise conclusion because nearly every experimenter explores a large range of metals thus compelling him to restrict the number of specimens of each. Moreover, at least in the less recent tests, the papers do not generally give sufficient descriptions of the chemical, metallurgical, metallographic, and mechanical properties of the metals tested.

To fill this lack, these tests were restricted to one grade of steel, typical of current manufacturing, a medium carbon steel (0.35 per cent carbon) processed in the electric furnace with utmost care, and aluminum killed to make it as stable as possible.

For the same reason, tests were confined to completely reversed loading in which $\sigma_{\min} = -\sigma_{\max}$. The purpose was to study:

1. The size effect in the various simple methods of loading, namely, tension-compression, alternate flexure, rotative flexure, and alternate torsion.
2. The effect of the shape of the specimen in alternate flexure, showing the comparison of results obtained with rectangular, circular, and square section specimens placed edgewise.
3. The effect of external notches of hyperbolic shape, as described by Neuber, on specimens stressed in tension-compression and reversed torsion.
4. The effect of the grain size of the metal for smooth and notched specimens stressed in tension-compression; three different grain sizes developed by appropriate heat treatments were used.
5. The effect of the polishing method on the results for smooth or notched specimens; numerous comparative tests were made on ground (and eventually

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 978.

mechanically polished) specimens and on electrolytically polished specimens.

Besides these main tests, alternate tension-compression tests were performed on flat specimens provided with holes of various diameters, in order to form an opinion on the theoretical ideas expressed concerning the effect of the stress gradient (see Appendix II).

DESCRIPTION OF TESTS

Steel:

The steel used was specially melted in an electric furnace from bloom scraps and was cast into cylindrical ingots 1.100 m in length by 0.275 m in diameter and weighing 580 kg (including the shrinkhead of 70 kg). A test ingot was cast for the preliminary tests. The ingots were rolled into round bars, 73 mm in diameter (furnace temperature, 1200 C; end of rolling, 1050 C). After rolling, 4.100 m, or 26.5 per cent, plus the shrinkhead were cut off from the top, and 2.200 m, or 14.5 per cent, were cut from the bottom of the bar (Fig. 1). The 9.100 m-long-bars were normalized at 725 to 750 C, and air cooled. They were bottom-end marked, and carefully checked and straightened: no flaws, seams, or scales were shown.

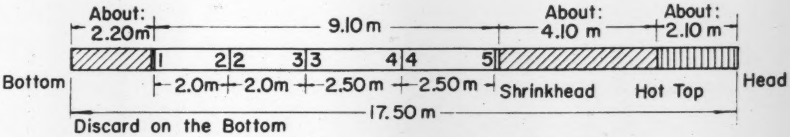

FIG. 1.—Steel Bar, 73 mm Diameter Rolled From Test Ingot.

The numbers 1 to 5, shown on the bar in Fig. 1, serve to locate their position in the ingot.

The chemical analysis of the test ingot, hammered into round bars 30 mm in diameter, showed the following composition, in per cent:

Carbon, per cent	0.380
Manganese, per cent	0.706
Silicon, per cent	0.256
Sulfur, per cent	0.011
Phosphorus, per cent	0.021

A spectrochemical analysis showed the following composition, in per cent:

Manganese, per cent	0.752
Silicon, per cent	0.227
Chromium, per cent	0.007
Molybdenum, per cent	0.008
Tin, per cent	0.010
Sulfur, per cent	0.054
Copper, per cent	0.063

The average from two chemical analyses of bars 73 mm in diameter, rolled from the bar numbered 4–5 (Fig. 1) showed the following composition in per cent:

Carbon, per cent	0.365
Manganese, per cent	0.725
Silicon, per cent	0.226
Sulfur, per cent	0.013
Phosphorus, per cent	0.019

The ends of each bar, examined by macrography, showed no crack, center porosity or marked segregation. The MacQuaid test showed an austenitic grain size corresponding to No. 6 (fine grain) of the ASTM scale.[3] Micrographs made, after etching, on the specimens from bars rolled and normalized showed that these bars had a regular structure in their whole section with fine grains of pearlite and boundaries of ferrite fairly marked (Fig. 2). A magnification of 100× showed approximately 95 grains per sq in., which corresponds to a grain size of 7 to 8 according to the ASTM scale.

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[3] Classification of Austenite Grain Size in Steels (E 19 – 46), 1955 Book of ASTM Standards, Part 1, p. 1602.

Two sets of bars of the steel described were subjected to two special heat treatments in order to obtain a medium grain structure (ASTM No. 4), and a coarse grain structure (ASTM No. 2).

To obtain the medium grain structure, the bar was heated to 1125 C and held for $2\frac{1}{2}$ hr, then furnace cooled for 5 hr to 650 C, then cooled in air. Under magnification of 100×, 11 grains per sq in. can be counted in the medium grained steel (Fig. 3).

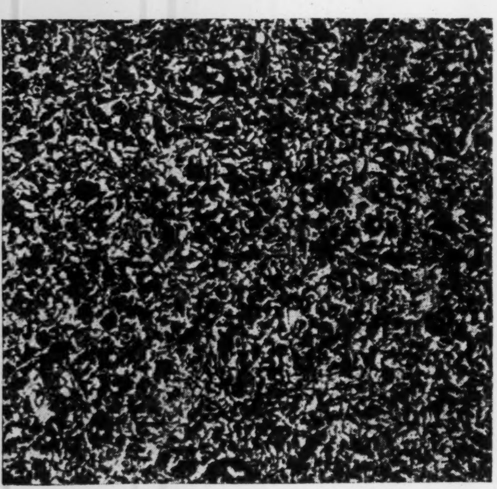

FIG. 2.—Micrograph Showing Fine Grain Structure of Specimens From Bars Rolled and Normalized (×100).

The coarse grained structure was obtained by heating the bar to 1150 C and holding for 2 hr then furnace cooling for 5 hr to 650 C. This structure shows a count of 2 or 3 grains per sq in. (Fig. 4).

The physical properties (Table I) of the fine, medium, and coarse grain steels were determined using 27 specimens, 13.8 mm in diameter (cross sectional area = 150 sq mm).

Since there are no major differences in the properties of the fine grain steel, according to the location of the bar in the ingot, the results obtained can be directly compared with all the fatigue specimens, without correction.

Data for the *S-N* curves (Fig. 5) was obtained with Baldwin type MD2 electronic equipment magnifying the elongations 20 times. Figure 5 gives the

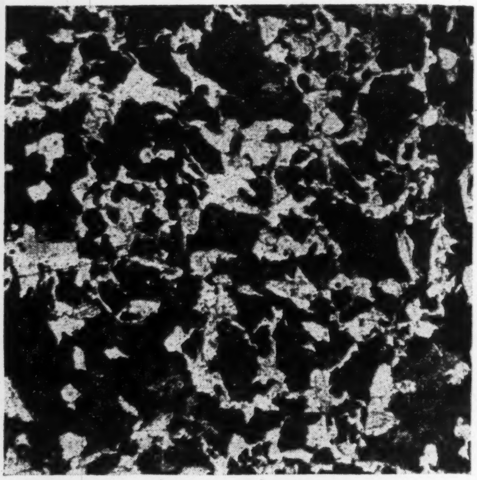

FIG. 3.—Micrograph Showing Medium Grain Structure of Specimens (×100).

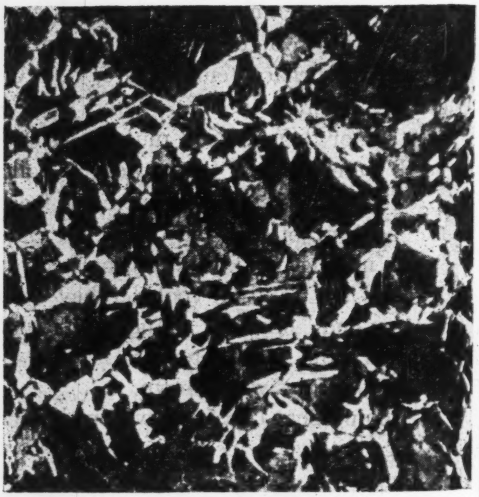

FIG. 4.—Micrograph Showing Coarse Grain Structure of Specimens (×100).

TABLE I.—PHYSICAL PROPERTIES OF FINE, MEDIUM, AND COARSE GRAIN STEELS.

Grain of Steel	Location in Ingot	Number of Tested Specimens	Lower Yield Strength, kg per sq mm[a]	Tensile Strength, kg per sq mm[c] avg.	Elongations, per cent		Reduction of Area, per cent	Elongation at End of Yield, per cent
					b	c		
Fine	1 - 2	6	29.8	56.4	30.4	25.2	60.1	0.81
	2 - 3	7	30.5	56.5	30.4	25.2	59.2	0.90
	3 - 4	5	33.9	58.5	28.4	23.5	58.1	1.20
Medium	1 - 2	4	31.1	62.2	24.9	21.4	47.9	0.70
Coarse	1 - 2	5	29.9	62.8	25.5	20.4	47.0	0.42

[a] 1 kg per sq mm = 1422 psi.

[b] Gage length = 5.65 $\sqrt{\text{cross-sectional area.}}$

[c] Gage length = 8.16 $\sqrt{\text{cross-sectional area.}}$

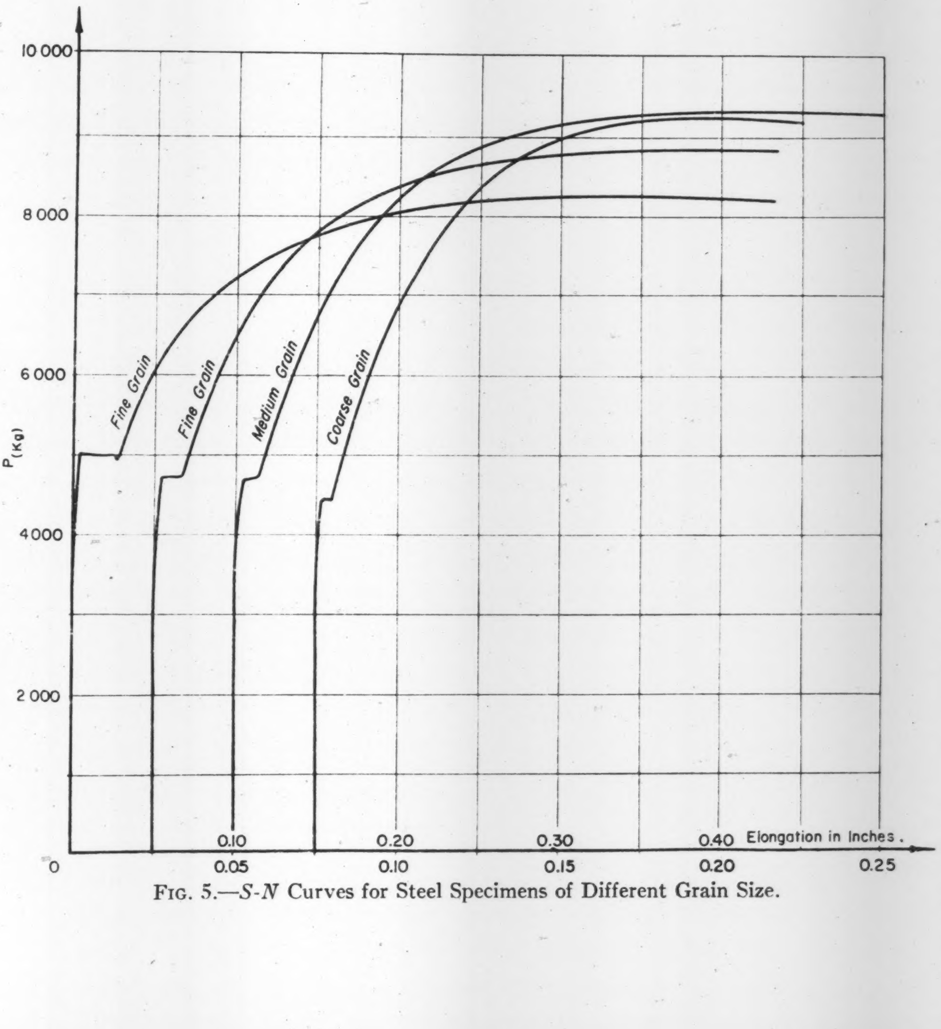

FIG. 5.—*S-N* Curves for Steel Specimens of Different Grain Size.

curves recorded from two fine-grain specimens, one medium, and one coarse-grain specimen. The average values for elongation, measured to within 0.001 in. at the yield point, are recorded in Table I. It can be seen that the heat treatments producing medium and coarse grains have effectually brought about an appreciable reduction of the length of the yield elongation.

Size Effect:

To study the size effect, dimensions in geometric progression with a common ratio of two were adopted in all series of tests. The minimum dimension compatible with the precision of the available machine and the maximum dimension compatible with the machine's power were determined for the whole series (Table II).

TABLE II.—SPECIMEN TYPE AND MACHINE USED.[a]

	Diameter, mm	Machine Used
Alternate Tension Compression	5	Baldwin-Sonntag SF-1-U
	8	Vibrophore Amsler Scale 10 ton
	16	
	32	Pulsating machine Amsler ± 25 ton
Alternate Flexure:		
(1) Specimens circular section	8	Baldwin-Sonntag SF-1-U
	16	
	23	
(2) Specimens rectangular section[b]	4 × 32	Baldwin-Sonntag SF-1-U
	8 × 16	
	16 × 8	
	32 × 4	
(3) Specimens square section on edge[c]	5	Baldwin-Sonntag SF-1-U
	10	
	20	
Rotative Flexure	2	Matra
	4	
	8	Schenck Simplex
	16[d]	Schenck UMBI
	32	
	56	
Alternate Torsion	8	Baldwin-Sonntag SF-1-U
	16	
	32	

[a] The tests were generally limited to 10 million cycles, except for some performed with the Amsler Vibrophore, which were carried on up to 30 million cycles. On the Matra machine, the specimen is stressed as a cantilever beam, whereas on the Schenck machine it is submitted to a constant moment. The specimens were not artificially cooled, except the smooth toric tension-compression specimens tested with Amsler Vibrophore which were fan-cooled.

[b] Length × width.

[c] Length of the side of the square.

[d] For these specimens, the machine was modified to stress the specimen as a cantilever beam and to afford greater precision in the range of the small bending moments.

EFFECT OF SHAPE OF SPECIMEN

According to von Philipp (**7**) and Weibull (**8**), there is an appreciable variation of the endurance limit depending upon the shape of the cross-section, which determines the volume of metal

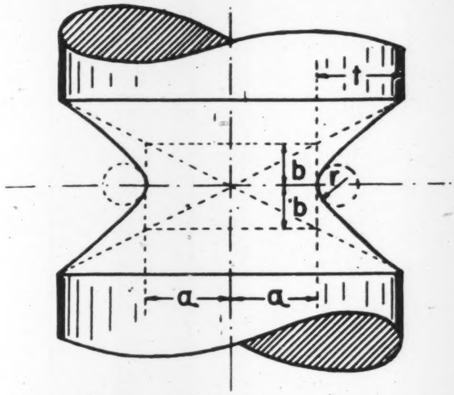

FIG. 6.—Shape of Notch in Round Specimens.

TABLE III.—SPECIMEN DIMENSIONS IN MILLIMETERS (SEE FIG. 7).

Notch	b	$\frac{a}{b} = \sqrt{\frac{a}{\rho}}$	$\frac{a^2}{b^2} = \frac{a}{\rho}$	ρ	t	$\frac{t}{\rho}$	$\sqrt{\frac{t}{\rho}}$
No. 1.........	5	1.6	2.56	3.125	8	2.56	1.6
No. 2.........	4	2	4	2	8	4	2
No. 3.........	2.667	3	9	0.889	6	6.75	2.6
No. 4.........	2	4	16	0.5	6	12	3.46
No. 5.........	1.6	5	25	0.32	4	12.5	3.54
No. 6.........	1.333	6	36	0.222	4	18.0	4.245
No. 7.........	1.0	8	64	0.125	4	32.0	5.66

submitted to the maxima stresses. In order to compare these theories with experience, comparative tests in alternate flexure were performed on bars of three types of sections: circular, rectangular, and square bent in the direction of a diagonal.

Notch Effect:

The notches had to possess the following properties:

1. Correspond accurately to known stress concentration factors and be capable of variation within large limits,

2. Allow calculation of the maximum stress gradient $\frac{d\sigma_{\max}}{dh}$ or even the gradient of maximum comparison stress according to the Hencky-Mises criterion $\frac{d\sigma_{c\ \max}}{dh}$, and

3. Be easily manufactured and permit optical control of dimensions after electrolytic polishing.

The simplest type of notch fulfilling these requirements appeared to be an exterior hyperbolic notch in a cylindrical specimen. In fact, the paper by Neuber (1) enables calculation of all the characteristics σ, σ_c, $\frac{d\sigma_{\max}}{dh}$, $\frac{d\sigma_{c\ \max}}{dh}$ of the stress state at the most stressed point.

The shape of the notch, Fig. 6, is defined by the ratio a/b of the semi-axis of the generating hyperbola. A series of 7 notches numbered from 1 to 7, in increasing sharpness, was used in the tests.

The radius, a, of the cross section of the specimens at the bottom of the notch is 16 mm for all the specimens. Table III shows the other two geometric characteristics b and ρ of the 7 notches. It also shows the depth, t, of the notches, which is necessary to calculate the stress concentration factor. The general shape of the specimen is shown in Fig. 7.

The stress concentration factor k_t was calculated by Neuber's formula (1). He gives the values of k_t for a notch of infinite depth, (k_t^f) as well as for a superficial notch made in a part of an infinitely great diameter (k_t^s); the real cases are situated between these two extremes; Neuber proposes to determine k_t by the interpolation formula:

$$\frac{1}{(k_t - 1)^2} = \frac{1}{(k_t^f - 1)^2} + \frac{1}{(k_t^s - 1)^2}$$

The values of k_t^f, k_t^s, and k_t calculated by these formulae, are given in Table IV. They have been checked by means of the charts of R. E. Peterson (6).

PREPARATION OF SPECIMENS

Eight specimens of each series were fabricated; this amount was considered generally sufficient to determine the endurance limit. However, several series of smooth specimens of rectangular section (Fig. 8) tested in alternate flexure contained 12 specimens. All the specimens were cut from round bars 73 mm in diameter. From the cross section of these bars, one, two or four specimens were taken according to the dimensions of the latter, so that the most stressed fibers of the specimens were approximately at the same average distance from the axis of the bar.

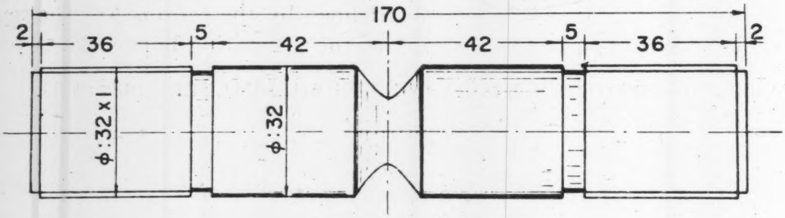

FIG. 7.—Notched Round Specimen.

TABLE IV.—STRESS CONCENTRATION FACTORS.

Stress	Tension			Flexure			Torsion		
Notch	k_t^f	k_t^s	k_t	k_t^f	k_t^s	k_t	k_t^f	k_t^s	k_t
No. 1	1.872	4.20	1.842	1.594	4.20	1.582	1.312	2.60	1.306
No. 2	2.28	5.00	2.257	1.844	5.00	1.824	1.432	3.00	1.423
No. 3	3.20	6.20	3.027	2.522	6.20	2.463	1.775	3.60	1.743
No. 4	4.14	7.92	3.86	3.255	7.92	3.150	2.050	4.46	2.004
No. 5	5.18	8.08	4.60	3.960	8.08	3.735	2.49	4.54	2.37
No. 6	6.18	9.49	5.415	4.690	9.49	4.40	2.852	5.245	2.70
No. 7	8.195	12.32	7.06	6.170	12.32	5.76	3.600	6.66	3.362

The shapes chosen for the smooth specimens depended on:

1. The method of gripping the specimen in the testing machine,

2. The necessity to avoid buckling of the tension-compression specimens tested with slow machines (Baldwin-Sonntag, 1500 cpm; Amsler ± 25 ton pulsating 500 cpm); consequently the calibrated part of the specimen was given a very dumpy toric shape.

3. The electric power limitations of the 10-ton Vibrophore; experience demonstrated that the steel used in the tests had a considerable dynamic hysteresis; the cylindrical specimens adopted in the first tests required, for maintaining the stress at a high frequency, a power rising above the maximum power of the Vibrophore and the specimens became very hot; therefore, toric specimens of the type shown in Fig. 9 were substituted.

After machining, all the specimens were ground with the grinding wheel placed in a manner to produce longitudinal scratches (practically without effect on the fatigue resistance). The toric shape of the cylindrical specimens facilitated the longitudinal grinding operation (Fig. 9).

The notches were machined with special hyperbolic profile tools, using a roughing tool and a finishing tool. The profiles were optically verified. The finishing tools were reground several times, the profile being checked each time. The machining was performed by cutting passes of decreasing depths in geometrical progression (1.00, 0.5, 0.25, and 0.1 mm) so as to have, after the last pass, a layer of strain-hardened metal as thin as possible.

After longitudinal grinding, most of the specimens were mechanically polished. The smooth specimens of cylindrical or toric shape were tested as ground, without supplementary preparation. The smooth specimens with rectangular or square section were hand-polished, using the set of metallographic papers 2, 1, 0, and 00 grits. The notched specimens were polished in the bottom of the notch in accordance with the method of the University of Illinois Polishing Procedure for Metallic Fatigue Specimens[4] except that the emery papers were replaced by a set of alumina powders of increasing fineness. The flat smooth (Fig. 8) or drilled specimens stressed in tension-compression were planed, then ground longitudinally. After drilling (holes 0.5 to 8 mm diameter) or boring (holes 16 to 32 mm diameter) the surface of the hole was polished by means of a revolving wooden mandrel provided with alumina powder. The edges of the hole were slightly rounded by this mandrel.

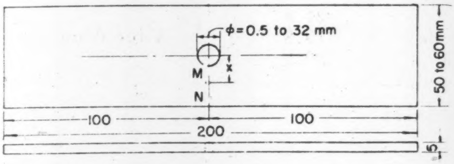

FIG. 8.—Drilled Specimen of Rectangular Section.

Some specimens were electrolytically polished after machining or grinding. The bath used was basically phosphoric acid; the electrodes were 18-8 stainless steel shaped as a tube for the smooth specimens and as a ring for the notched ones. The electro-chemical etching was continued until the last scratch of machining disappeared. Measurements of diameter made before and after the polishing give 0.10 mm as an average for the layer of metal taken off by electrolysis.

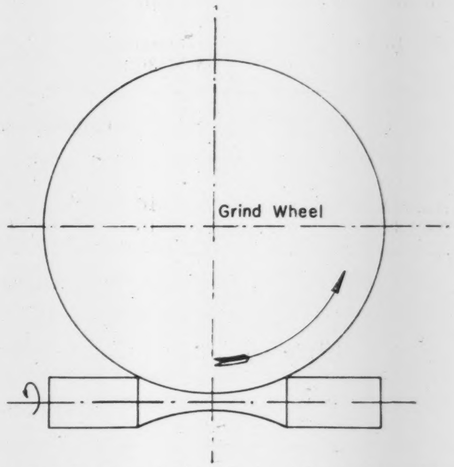

FIG. 9.—Longitudinal Grinding Operation of Toric Specimen.

The diameter of the notched specimens after polishing was measured by means of a Zeiss projection apparatus; and on the same apparatus the shape of the notches was checked against standard profiles. Those specimens failing to conform were discarded. All those specimens with the No. 7 notch (the sharpest) were ultimately discarded because the profile showed excessive variation due to the electrolytic polishing operation.

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[4] Fatigue Manual (Issued as separate publication *ASTM STP No. 91*).

TABLE V.—SMOOTH SPECIMENS.

ALTERNATE TENSION-COMPRESSION

Test	Diameter, mm	Grain	Endurance Limit, kg per sq mm	Machine
		MECHANICAL POLISHING		
No. 2	5	Fine	24.2	Baldwin
No. 3	6		25.0	
No. 4	16		23.7	Vibrophore
No. 5	32		22.9	Amsler ± 25
No. 25	16	Medium	22.4	Vibrophore
No. 34	16	Coarse	20.9	
No. 43	rectangular 5 × 30 mm	Fine		Vibrophore
		ELECTROLYTICAL POLISHING		
No. 18	16	Fine	21.8	Vibrophore
No. 30	16	Medium	19.8	
No. 39	16	Coarse	20.2	

ALTERNATE FLEXURE

Test	Shape	Dimensions, mm	Endurance Limit, kg per sq mm
	MECHANICAL POLISHING		
No. 1	rectangular	4 × 32	25.2
No. 2	rectangular	8 × 16	26.4
No. 3	rectangular	16 × 8	24.5
No. 4	rectangular	32 × 4	25.3
No. 5	square on edge	5 × 5	27.6
No. 6	square on edge	10 × 10	27.6
No. 7	square on edge	20 × 20	25.3
No. 8	circular	8 diam	27.7
No. 9	circular	16 diam	27.5
No. 10	circular	23 diam	27.2
	ELECTROLYTICAL POLISHING		
No. 14	circular	8 diam	24.4
No. 15	circular	16 diam	25.0
No. 16	circular	22 diam	23.2

TABLE V—*Continued*

Rotative Flexure

Test	Diameter, mm	Endurance Limit, kg per sq mm
Mechanical Polishing		
No. 1	2	28.6
No. 2	4	27.6
No. 3	8	26.2
No. 4	16	24.7
No. 5	32	22.4
No. 6	56	23.0
Electrolytical Polishing		
No. 7	2	23.4
No. 8	4	22.5
No. 9	8	21.2
No. 10	16	20.7
No. 11	32	22.1

Alternate Torsion

Test	Diameter, mm	Endurance Limit, kg per sq mm
Mechanical Polishing		
No. 1	16 (toric)	15.7
No. 2	16 (cylindric)	14.3
No. 3	32	13.3
Electrolytical Polishing		
No. 10	16	14.1

RESULTS OF THE TESTS

The results obtained on smooth specimens are shown in Table V. The results obtained on cylindrical specimens provided with hyperbolic notches are listed in Table VI. Finally the results obtained with flat bored specimens are recorded in Table VII.

Results:

A preliminary observation must be made: the electrolytically polished specimens give generally more uniform results than the mechanically polished ones; this is clearly apparent by comparing the corresponding Wöhler curves, the dispersion of which is smaller in the case of the electrolytic polishing.

The mechanical polishing leaves an altered layer, the properties of which depend on the mode of polishing and are therefore different from one laboratory to another. But, X-ray measurements have demonstrated, according to Hempel (**15** p. 483), that the depth of the altered superficial layer due to machining may vary from 0.02 to 0.05 mm. As a matter of fact, measurements show that the layer dissolved by electrolysis is normally greater than 0.07 mm. Then it can be stated that the electrolytically polished specimens were virgin metal.

The use of electrolytic polishing in fundamental research on fatigue of metals is strongly advocated. It can be argued that this condition of the surface is never realized in practice; however, it is a state of reference well defined and reproducible with precision. In fatigue research it might conceivably occupy a place similar to the simply supported bar in the buckling tests; the simple support is not realized in the industrial columns any more than electrolytic polishing is in the machine parts.

Table VI shows that the size effect is slight in alternate tension-compression. This concurs with Buchmann's observation for other metals. The slight differences observed must probably be ascribed to a frequency effect. The Vibrophore, which works at a frequency of about 8500 cpm, gives results superior on an average by 4 per cent to those obtained with the Baldwin machine, which makes 1500 cpm, or with the Amsler machine ±25 ton which makes 500 cpm.

The effect of size was small in alternate flexure, where it follows similar laws for the specimens with rectangular, circular, or square on edge sections.

The size effect is obvious in rotative flexure, where the difference between the endurance limits of specimens 2 and 32 mm in diameter reaches 28 per cent. The plot of the results obtained with specimens of transverse dimensions growing in geometric progression shows a curve nearly linear (Fig. 10) for the mechanically polished as well as for the electrolytically polished specimens. Therefore, the size effect in rotative flexure follows an approximately logarithmic law.

This law is not indefinitely applicable but it is probably related to a law, $R_f =$ constant, for a diameter of about 60 mm; in fact Fig. 10 shows that the endurance limit by rotative flexure of 32-mm specimens is nearly equal to the endurance limit by tension-compression.

The laws observed for the two types of polishing are nearly parallel. Consequently, there is an appreciable size effect on virgin metal specimens that cannot be ascribed to a superficial strain-hardened layer formed by machining. On this particular point results confirm the conclusion of Morkovin and Moore (**9**). However, the 2, 4 and 8-mm diam specimens have a toric profile, whereas the 16, 32, and 56 mm diam specimens have a cylindrical shape; and since the endurance limit is always greater for

TABLE VI.—SPECIMENS PROVIDED WITH EXTERIOR HYPERBOLIC NOTCHES.

	Test	Grain	Notch	Endurance Limit, kg per sq mm	k_f	k_t	$q = \frac{k_f - 1}{k_t - 1}$
MECHANICAL POLISHING							
Alternate tension-compression	No. 6	Fine	No. 1	14.0	1.692	1.842	0.821
	No. 7		No. 2	12.2	1.94	2.257	0.812
	No. 8		No. 3	11.1	2.13	3.027	0.560
	No. 9		No. 5	8.8	2.69	4.60	0.470
	No. 10		No. 7	9.30	2.55	7.06	0.256
	No. 26	Medium	No. 1	15.7	1.42	1.842	0.499
	No. 27		No. 2	13.6	1.65	2.257	0.515
	No. 28		No. 4	8.7	2.57	3.86	0.551
	No. 29		No. 7	10.9	2.06	7.06	0.174
	No. 35	Coarse	No. 1	14.2	1.465	1.842	0.552
	No. 36		No. 2	12.2	1.707	2.257	0.563
	No. 37		No. 4	8.0	2.51	3.86	0.528
	No. 38		No. 7	9.5	2.20	7.06	0.198
Alternate flexure	No. 11	Fine	No. 7	15.7	1.75	7.06	0.124
Alternate torsion	No. 4	Fine	No. 1	13.7	1.044	1.306	0.164
	No. 5		No. 2	12.2	1.019	1.423	0.409
	No. 6		No. 3	10.8	1.173	1.743	0.436
	No. 7		No. 4	11.9	1.324	2.004	0.200
	No. 8		No. 6	11.9	1.201	2.70	0.118
	No. 9		No. 7	11.6	1.232	3.36	0.098
ELECTROLYTICAL POLISHING							
Alternate tension-compression	No. 19	Fine	No. 1	12.9	1.69	1.842	0.820
	No. 20		No. 2	12.1	1.80	2.257	0.637
	No. 21		No. 3	10.1	2.16	3.027	0.572
	No. 22		No. 4	8.8	2.48	3.86	0.518
	No. 23		No. 5	8.4	2.60	4.60	0.445
	No. 24		No. 6	7.9	2.76	5.42	0.398
	No. 31	Medium	No. 1	13.7	1.445	1.842	0.528
	No. 32		No. 4	8.0	2.48	3.86	0.518
	No. 33		No. 6	6.75	2.94	5.42	0.440
	No. 40	Coarse	No. 1	13.85	1.46	1.842	0.545
	No. 41		No. 4	8.5	2.38	3.86	0.483
	No. 42		No. 6	7.5	2.69	5.42	0.383
Alternate flexure	No. 12	Fine	No. 1	15.25	1.65	1.824	0.790
	No. 13		No. 4	13.0	1.93	3.735	0.340
Alternate torsion	No. 11	Fine	No. 1	12.8	1.100	1.306	0.327
	No. 12		No. 2	12.0	1.175	1.423	0.414
	No. 13		No. 3	11.0	1.280	1.743	0.377
	No. 14		No. 4	11.4	1.235	2.004	0.234
	No. 15		No. 5	12.8	1.100	2.37	0.073

TABLE VII.—BORED SPECIMENS STRESSED IN ALTERNATE TENSION COMPRESSION.

Test	Radius of Hole, mm	Endurance Limit, kg per sq mm	k_f	k_t	q
No. 44......	0.25	14.8	1.60	2.98	0.303
No. 45......	0.5	13.7	1.73	2.96	0.372
No. 46......	1.0	13.1	1.81	2.92	0.423
No. 47......	2.0	12.3	1.93	2.88	0.495
No. 48......	4.0	11.8	1.99	2.77	0.560
No. 49......	8.0	13.2	1.80	2.56	0.513
No. 50......	16.0	14.0	1.69	2.24	0.556

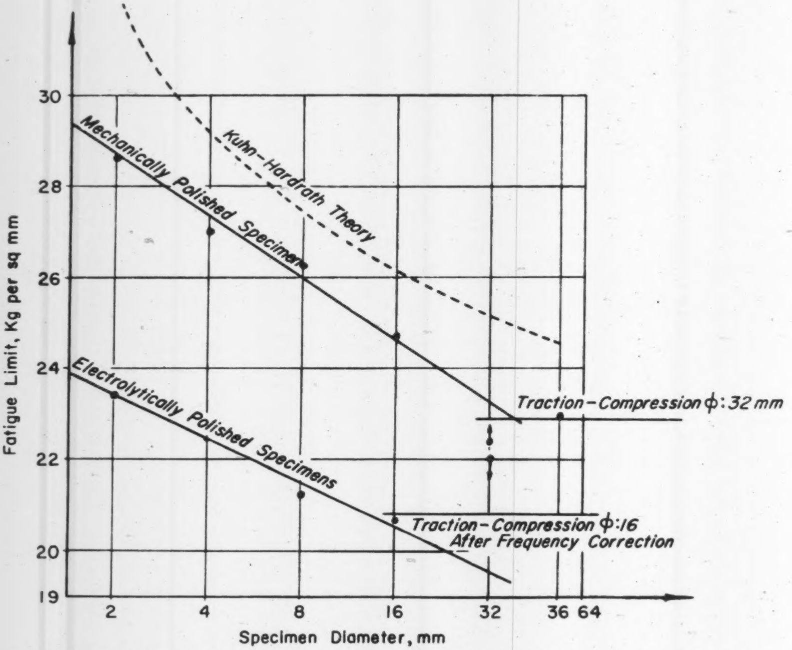


FIG. 10.—Size Effect in Rotative Flexure for Mechanically and Electrolytically Polished Specimens

toric than for cylindrical specimens the real size effect is certainly less than indicated in the results of tests.

Tests seem to demonstrate that the size effect in rotative flexure is fundamentally different from the one existing in alternate flexure (Table VIII). The former seems therefore to ensue at least partially from the rotative character of the test and might perhaps be explained by the existing out-of-phase between the cyclic dilatations imposed upon the metal during the test and the resulting tensions, because of the existence of an hysteresis loop (Fig. 11). As to the torsion tests, the results obtained are too dispersed and insufficient to draw definite conclusions. The results obtained are not in good agreement with the theory of von Philipp (7). They do not agree very well with the formula proposed by Kuhn and Hardrath (11):

$$S_{RB} = S_{AL}\left(1 + \sqrt{\frac{A}{r}}\right)$$

where A is the constant of Neuber and r the radius of the specimen. In fact, if the average value for A obtained by these tests were adopted, the disagreement would be complete. If the value of A, deduced from Fig. 3 of the Kuhn and

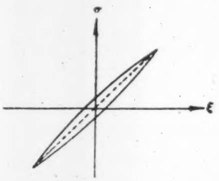


FIG. 11.—Hysteresis Loop in Rotative Flexure Tests.

TABLE VIII.—VALUES OF ENDURANCE LIMITS, σ_f.

Polishing	Stress	8-mm diam	16-mm diam	32-mm diam
Mechanical...............	Rotative Flexure	26.2	24.7	22.4
	Alternate Flexure	27.7	27.5	25.2
Electrolytical.............	Rotative Flexure	21.2	20.7	22.1
	Alternate Flexure	24.4	25.0	23.2

Hardrath paper (11) were adopted, and if the endurance limit in alternate tension-compression S_{AL} were used, the resulting curve would be the dotted line shown in Fig. 11. This curve does not agree well with the results of this paper; by changing the value of S_{AL}, the curve would be lower but its shape would remain different from the logarithmic law experimentally obtained.

The fundamental difference existing between the rotative flexure stresses and the alternate flexure ones, is that in the former all the external fibers of the specimen are stressed successively in each cycle, whereas in the latter, the same fibers always undergo the maxima stresses. The volume strongly stressed is much smaller in alternate flexure.

According to the theory of Weibull, based on the statistical distribution of defects, the endurance limit should be higher in alternate flexure. Table VIII shows that experience confirms these theoretical provisions; however, a quantitative control of the theory of Weibull would require an enormous number of experiments and is therefore impossible.

The effect of the shape of the specimen has been studied only in alternate flexure, the results obtained being recorded in Table IX. These results show a slight increase of the endurance limit from rectangular to circular to square on edge section, which is still in accordance with the theories of Weibull. However, the differences observed are small and of the same order as the precision of the measurements; so it is not possible to draw any quantitative conclusion. It would be necessary, in order to determine the trend more definitely, to carry on new tests on many specimens, eliminating the disturbing influence of the mechanical polishing by a systematic use of electrolytically polished specimens.

It has been stated in many papers that the value of the ratio τ_f to σ_f of endurance limits in torsion and bending, respectively, experimentally determined is in accordance with the criterion of Hencky and von Mises, which states that this ratio should be $1/\sqrt{3} = 0.577$.

The quantity τ_f can be determined

TABLE IX.—VALUES OF ENDURANCE LIMITS, σ_f.

Dimension in millimeter (for definition see Table III)	h.....	4	8	16	32
	d.....	8	16	23	..
	c.....	5	10	20	..
Shape of Section:					
Rectangular...........		25.2	26.4	24.5	25.3
Circular...............		27.7	27.5	25.2	..
Square on edge.........		27.6	27.6	25.3	..

only by a torsion test, which then coincides with the endurance limit in alternate torsion. The quantity σ_f, on the contrary, can be determinated in tension-compression, in alternate flexure, or in rotative flexure. In Table X are recorded the values of the ratio τ_f/σ_f calculated by the various possible methods. Table X shows that the ratio may vary within large limits, and is approximately $1/\sqrt{3}$ only if σ_f represents the endurance limit of tension-compression or rotative flexure, but not if σ_f is determined in alternate flexure.

In brief, it is certain that the state of surface of the specimens mechanically polished, cylinder or toric shaped, and their method of stressing (constant or linearly variable bending moment) play an important part in the results hitherto published about the size effect. Consequently, it is impossible to draw any positive conclusion from these tests. However, we cannot completely agree with Hempel (16) when he states that the size effect is mostly of technological origin, for these tests indicate a size effect of appreciable magnitude on electrolytically polished specimens.

TABLE X.—VALUES OF RATIO τ_f/σ_f.

Polishing		Mechanically Polished		Electrolytically Polished
Diameter, mm		16	32	16
Comparison Stress	Tension-Compression	0.603	0.571	0.647
	Alternate Flexure	0.520	0.528	. . . . .
	Rotative Flexure	0.578	0.594	0.631

RESULTS FROM TESTS ON NOTCHED SPECIMENS

The first conclusion of the tests on notched specimens is particularly clear: there is a fundamental difference between the results of the mechanically polished notched specimens and those electrolytically polished (see Table VI).

The endurance limit R_f of electrolytically polished specimens progressively decreases when the sharpness of the notch increases; while the endurance limit of the mechanically polished specimens goes down to a minimum, and then rises again. This fact is characteristically repeated for the three grain-sizes under observation. The meaning of these results can be deduced by imagining that the hyperbolic tool causes the metal of the specimen to be reinforced by strain hardening up to a certain depth. This reinforcing effect, similar to that obtained in shot-peened specimens, results in compensating, then outweighing the damaging effect of the geometrical shape of the notch. It seems necessary, therefore, in order to progress in the fundamental knowledge of the notch effect, to observe this effect on virgin specimens, that is, electrolytically polished. This shows also that it is hopeless to draw precise conclusions from the countless data on notched parts published in the literature, because there is practically no information on the parameter "surface state," the role of which is outstanding.

Notched Specimens Stressed by Tension-Compression:

The author has compared his experimental results with the formulae of

Neuber (1) and Heywood (3); in both cases there is hardly any agreement (see Appendix I). The author believes these formulae are not appropriate for the steel under study. However, the comparison with the curve given by Peterson for the normalized steels (Fig. 12) is in good agreement with the results on fine grain, notched, mechanically polished specimens. Therefore, it seems that this curve is actually the best base to adopt for designing notched machine parts.

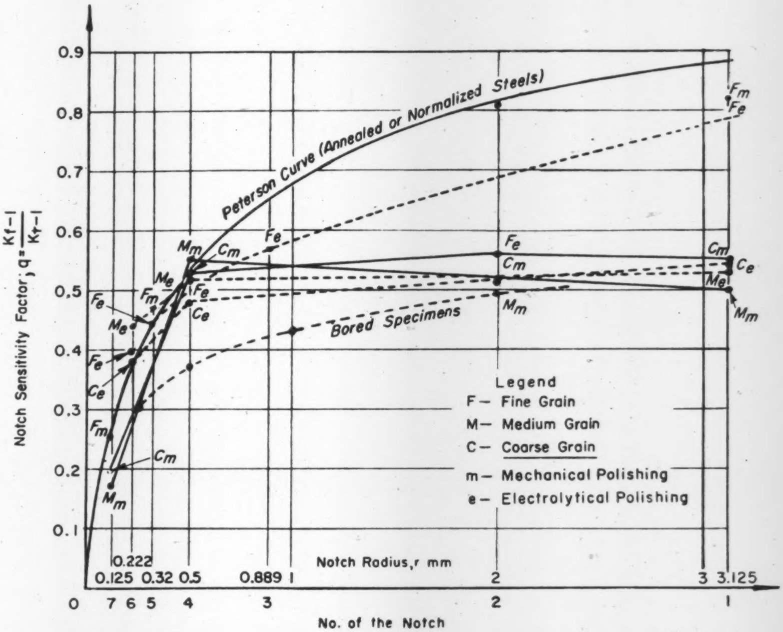


FIG. 12.—Notch Sensitivity Factor for Fine, Medium, and Coarse Grain Specimens Both Mechanically and Electrolytically Polished.

The only drawback of the Peterson curve is that it does not allow association of the size effect to the notch effect. In fact, in a smooth part stressed in rotative flexure:

$$\sigma = \frac{M_y}{I}\ ; \qquad \text{thus:} \quad \frac{d\sigma}{dy} = \frac{M}{I}\ ,$$

and

$$\frac{1}{\sigma_{\max}}\frac{d\sigma_{\max}}{dy} = \frac{1}{r}\ ; \qquad \text{also,} \qquad k_t = 1.$$

But the specimen cannot be assimilated to a notched specimen with the reduced gradient heretofore, because, if $k_t = 1$, we must have also $k_f = 1$, unless $q = \frac{k_f - 1}{k_t - 1}$ should be infinite. On the contrary, a law of the kind

$$\frac{k_f}{k_t} = f\left(\frac{1}{\sigma_{\max}}\frac{d\sigma_{\max}}{dy}\right)$$

could still be applied in the limiting case where:

$$k_t = 1 \qquad \text{and} \qquad \frac{1}{\sigma_{\max}}\frac{d\sigma_{\max}}{dy}\frac{1}{r}$$

and would give a value:

$$k_f = \frac{\text{endurance limit in alternative tension compression}}{\text{endurance limit in rotative flexure.}}$$

$$= \text{function of } \left(\frac{1}{r}\right) \text{ inferior to 1.}$$

The only laws of this type which have been proposed according to the author's information, are that of Heywood (**4**, p. 351):

$$\frac{k_f}{k_t} = \frac{1}{1 + 2\sqrt{\frac{a}{r}}}$$

and the one of Bollenrath and Troost (**17**):

$$\frac{k_f}{k_t} = 1 - \frac{\frac{1540}{R_r}}{\frac{1}{1 + \frac{R_r}{13\ 700}} + r}$$

where:

R_r = static tensile strength in kg per sq cm;

r = radius of curvature at the bottom of the notch in cm.

Unfortunately, Heywood's law does not perfectly agree with the author's results (see Appendix I). As for the one of Bollenrath and Troost, it does not agree at all with the author's results, nor, with those obtained by other experimenters. As a matter of fact, the tests point out that, when the radius, r, of the notch decreases, the notch sensitivity, q, decreases and approaches zero; that is to say k_f should approach 1, which does not occur with the above formula.

The plotted results of tests on fine grain electrolytically polished specimens fall under the curve of Peterson, thereby illustrating that electrolytic polishing results in slightly decreasing the notch sensitivity of the metal.

Finally, for the four series of medium or coarse grain notched specimens (Fig. 12) either mechanically or electrolytically polished, the notch sensitivity factor of the metal is nearly constant in the great interval, $0.5 \leq r \leq 3.125$ mm, and is equal to 0.53 ± 0.03. This conclusion alone summarizes 14 endurance limits, that is the results of tests of 112 specimens.

Notched Specimens Stressed in Alternate Torsion:

When the sharpness of the notch is increased, the endurance limit decreases, reaches a minimum, then rises again. This fact appears on electrolytically polished specimens as well as on mechanically polished ones. This must be attributed to the strain hardening brought about by the tool cutting the notch, this strain hardening being too deep to be eliminated by the dissolution of the superficial layer which ensues from the electrolytical polishing. The curves $q = f(r)$ are shown in Fig. 13; they do not tend towards 1 when r increases. Also the notch sensitivity in torsion is definitely smaller than in tension-compression.

Finally, the design of the curve $q = f(r)$ depends largely on the shape of the specimen by means of which the endurance limit of the smooth part has been determined. The curve for the mechanically polished cylindrical specimens is shown in full line, and the electrolytical polished in dotted line. But, for a toric specimen, the endurance limit of which is higher (15.7 kg per sq mm instead of 14.3 kg per sq mm), the notch sensitivity becomes higher, as shown by the dots in Fig. 13.

Bored Specimens Stressed in Tension Compression:

The results of tests on smooth bored specimens (Table VII) give a curve

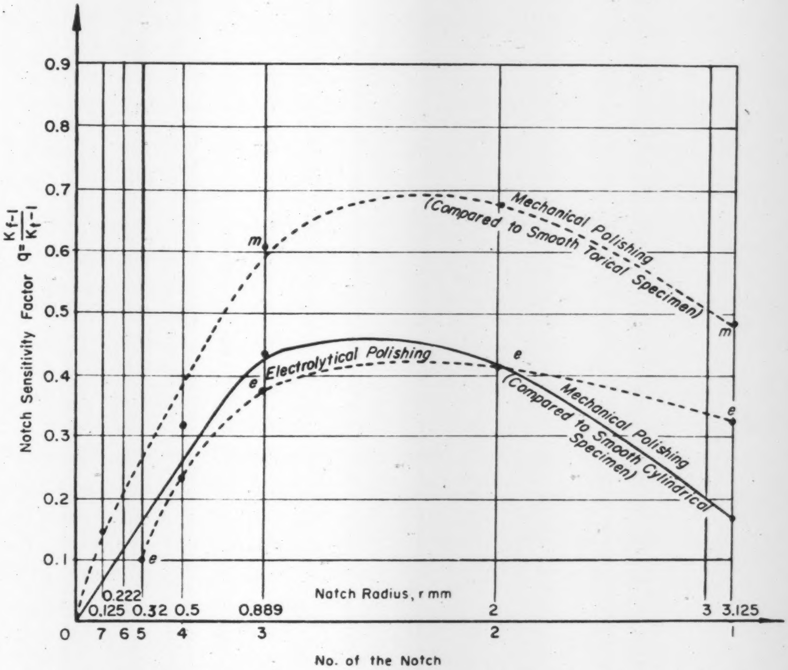


FIG. 13.—Notch Sensitivity Factors for Specimens Stressed in Alternate Torsion.

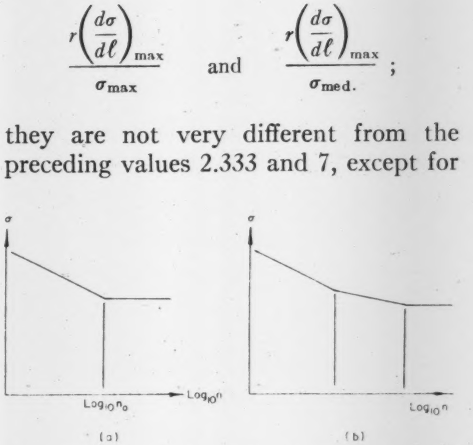
$q = f(r)$. The value 23.7 kg per sq mm, for smooth torical specimens was adopted as the endurance limit for smooth specimens. A plot of the experimental points gives the dotted curve, Fig. 12, situated much below the curve of Peterson.

Peterson plots the radius, r, along the abscissa, while according to Siebel (12) and Ros (13) the notch sensitivity factor should depend on the gradient of stress. The author could believe that the discordance between the dotted curve and Peterson's curve is due to this fact, but will show that this conclusion is wrong.

The gradient $\left(\frac{d\sigma}{dl}\right)_{\max}$, or the quantity $\frac{1}{\sigma_{\text{medium}}}\left(\frac{d\sigma}{dl}\right)_{\max}$ or the quantity $\frac{1}{\sigma_{\max}}\left(\frac{d\sigma}{dl}\right)_{\max}$, may be adopted as the parameter. For a bored part of infinite width $\left(\frac{d\sigma}{dl}\right)_{\max} = \frac{7\sigma_{\text{med}}}{r}$; then $\frac{1}{\sigma_{\text{med}}}\left(\frac{d\sigma}{dl}\right)_{\max} = \frac{7}{r}$; and $\frac{1}{\sigma_{\max}}\left(\frac{d\sigma}{dl}\right)_{\max} = \frac{7}{3r} = \frac{2.333}{r}$. Table XV gives the values of the quantities

$$\frac{r\left(\frac{d\sigma}{d\ell}\right)_{\max}}{\sigma_{\max}} \quad \text{and} \quad \frac{r\left(\frac{d\sigma}{d\ell}\right)_{\max}}{\sigma_{\text{med.}}};$$

they are not very different from the preceding values 2.333 and 7, except for



(a) (b)

FIG. 14.—Reduced Gradient Effect.

TABLE XI.—VALUES FOR REDUCED GRADIENT.

Notch	r, mm	$\frac{\left(\frac{d\sigma}{dl}\right)_{\max}}{\sigma_{\text{med}}}$	$\frac{\left(\frac{d\sigma}{dl}\right)_{\max}}{\sigma_{\max}}$	$\frac{2}{r}$
No. 1	3.025	1.175	0.628	0.661
No. 2	2.000	2.215	0.971	1.000
No. 3	0.889	7.17	2.24	2.25
No. 4	0.5	16.31	4.07	4.000
No. 5	0.320	32.7	6.31	6.25
No. 6	0.222	56.1	9.09	9.000
No. 7	0.125	132	16.10	16.00

TABLE XII.—RESULTS ON TORIC AND ELONGATED SPECIMENS IN ROTATIVE FLEXURE.

Longitudinal Shape of Specimen	Mechanical Polishing	Electrolytical Polishing
Toric (Fig. 10)	26.2	21.2
Cylindrical very elongated . .	23.8	19.0

the holes of 8 and 16 mm radius. The calculations for the preceding gradients for a tension tested part having a hyperbolic notch of infinite depth, were based on the strict formulae of Neuber (1).

The results obtained are recorded in Table XI. The values found for the reduced gradient $\frac{1}{\sigma_{\max}}\left(\frac{d\sigma}{dl}\right)_{\max}$ are very near to those given by the approximate expression $\frac{2}{r}$, which is proposed by Petersen (10). Consequently, if the parameter on which the notch sensitivity factor q depends is $p = \frac{1}{\sigma_{\max}}\left(\frac{d\sigma}{dl}\right)_{\max}$, this parameter has practically the same expression for the two types of specimens $\left(\frac{2}{r} \text{ against } \frac{2.33}{r}\right)$ and no important error is made by plotting, r, as abscissae instead of the said parameter. If, on the contrary, one adopts, as parameter on which q depends, the quantity:

$$p' = \frac{1}{\sigma_{\text{med}}}\left(\frac{d\sigma}{dl}\right)_{\max}$$

it can be seen that the radii r_{eq} of the hyperbolic notches corresponding to the radii of the holes are very different from r. In order to find these equivalent radii r_{eq}, the curves $p = f\left(\frac{1}{\rho}\right)$ are drawn for the bored parts and the hyperbolic notched parts. The equivalent radii can be directly read on the graph; they are recorded in the last column of Table XV.

Plotting as ordinates, Fig. 13, the values for q obtained experimentally (recorded in Table VI) and as abscissae the equivalent radii, as shown in Table XV, the result is a curve (not drawn) which would fall further from the curve of Peterson than the dotted curve.

It can be concluded from the preceding argument that, whatever the definition chosen for the gradient, p, the curve $q = f(p)$ of the bored specimens considerably differs from the one corresponding to the revolution hyperbolic notches. This difference seems to point out that Peterson's curve cannot be applied to all types of notches.

The author has also compared his results with the theory proposed by Roš (13); the principal practical consequences of this theory are as follows:

(*a*) The stress gradient has no influence on the fracture by fatigue and k_f is a definite function of k_t;

(*b*) One must associate with the specimens stressed by rotative or alternate flexure and alternate torsion coefficients of stress concentration respectively equal to $k_t = 1.7$ and $k_t = 1.33$.

These values correspond, through the use of the curve $k_f = f(k_t)$, to determined values of k_f; consequently the endurance limits in rotative or alternate flexure and in alternate torsion should be a constant fraction of the ones obtained in alternate tension-compression.

The author believes that the results reported in this paper definitely negate the hypothesis of Roš; in fact, the tests on bored specimens show an unquestionable effect of gradient. On the other hand, the points representing the tests on smooth specimens do not correctly take place on the curve $k_f = f(k_t)$ obtained with tests on notched specimens.

The principal reason for this is that the points representing the tests on smooth specimens tested by rotative or alternate flexure spread out upwards on account of the evident size effect observed in these tests, while Roš states that this size effect does not exist. Besides, the theory of Roš puts on the same level the rotative flexure and the alternate flexure, while results obtained for these two stresses are definitely different. Incidentally, Roš explains the fatigue behavior of notched specimens by the progressive development, in the bottom of the notch, of residual stresses of compression during the fatigue test.

The "Centre National de Recherches Métallurgiques," Section de Liège, performed measurements of residual stresses by X-ray diffraction on some specimens. On each specimen, several measurements were made after numbers of stress cycles increasing approximately in geometrical progression. These measurements have not detected any noticeable residual stress, but their precision was unfortunately very low. It would be very interesting to resume these measurements with improved means and with a steel that would enable obtaining the maximum accuracy.

Notwithstanding, the important differences between the results for the mechanically polished specimens and those for the electrolytically polished ones demonstrate that the layer of metal strain hardened by machining and subsequent polishing has a considerable effect on the behavior of the specimen.

In the author's opinion, this effect by far outweighs the one of the residual compression stresses, the real existence of which remains to be demonstrated.

Effect of Grain Size:

The results obtained show that the notch sensitivity factor, q, decreases very much, for the slight notches, when one passes from the fine grain ($q_{\text{mean}} = 0.8$) to the medium grain ($q_{\text{mean}} = 0.53$); on the contrary, there is no appreciable difference between medium and coarse grain.

This points out that the "structural elementary unity" of the steel, at least if this concept makes sense, is greater for a medium or coarse grain steel than for a fine grain steel. However, this structural unit does not coincide with the medium dimension of the grain micrographically determined.

The dispersion of the results of fatigue tests is definitely greater for the medium or coarse grain grades than for the fine grain grade.

ADDITIONAL INFORMATION DEDUCED FROM THE TESTS

Although the tests were mostly made for the study of the endurance limit, conclusions which may be drawn in the domain of the finite lives of the specimens should be examined.

The Wöhler curves all presented the classical form composed of two straight lines, except for the notched specimens stressed by alternate torsion, which all show a polygonal Wöhler curve composed of three straight lines.

In the course of the tests, the author has observed the effect of the longitudinal shape of the specimens on their resistance to rotative flexure; and has effectively obtained results clearly different on specimens of toric shape and on specimens of very elongated cylindrical shape, ($l/d = 20$) as shown in Table XII.

Similarly, the torsion tests on the two types of specimens have given definitely different results (15.7 kg per sq mm against 14.3 kg per sq mm).

This difference can be explained by the theory of Weibull (8) and we intend to investigate the matter more thoroughly by tests on diversely shaped specimens electrolytically polished and by multiple tests in order to obtain better average values.

SUMMARY

Results of these tests:

1. Demonstrate the importance of electrolytic polishing in the fundamental researches on the nature of the fatigue phenomenon.
2. Emphasize in an unquestionable way an important size effect in rotative flexure and furnish the quantitative law of variation of the corresponding endurance limit.
3. Show practically no size effect in tension-compression and slight in alternate flexure.
4. Permit selection, among the many theoretical laws about fatigue resistance of notched parts, of the law of Peterson, which shows the best agreement with the experimental facts.
5. Provide diverse curves useful for designing machine parts made of medium carbon steel.
6. Emphasize the effect of the metallographical grain size, which results in largely reducing the notch sensitivity and stabilizing the value of this sensitivity by annulling, for the shallow notches, the effect of the stress gradient.
7. Finally, point out that the stress gradient has an appreciable effect on the fatigue resistance.

These tests provide therefore, for the studied medium carbon steel, a partial answer to unsolved problems which were brought to the attention of engineers in an ASTM Special Technical Publication (14).

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Our colleague, Mr. F. Campus, prorector of the University, who permitted us to use the Amsler ±25 ton fatigue machine of his laboratory to perform a series of tests. The author is also grateful to Mr. E. Bodart, for the care with which the machine tools for the hyperbolic notches were constructed and for the optical control of these notches after electrolytic polishing.

APPENDIX I

The ability of the Neuber and Heywood formulae to represent the results of these tests has been appreciated as follows:

1. *Neuber formula*

This formula is written ((1), p. 163):

$$q = \frac{k_f - 1}{k_t - 1} = \frac{1}{1 + \sqrt{\frac{A}{r}}}$$

where:

r is the radius of curvature at the bottom of the notch and A a characteristic constant of the material (for a definite heat treatment).

Kuhn and Hardrath have suggested the hypothesis that A depends only on the static tensile strength R_r of the material; they have deduced from tests published in the literature, a curve $\sqrt{A} = f(R_r)$.

In order to test the Neuber formula, it can be stated that it may be written:

$$\left(\frac{1 - q}{q}\right)^2 r = A,$$

TABLE XIII.—AVERAGE AND EXTREME VALUES OF "A" UNDER TENSION, COMPRESSION, AND ALTERNATE TORSION.

Stress	Grain	Mode of Polishing	Average A, mm	Extremes Values of A
Tension Compression . . .	Fine	Mechanical	0.454	0.108– 1.057
		Electrolytical	0.463	0.151– 0.688
	Medium	Mechanical	2.140	0.330– 3.160
		Electrolytical	1.100	0.360– 2.510
	Coarse	Mechanical	1.43	0.400– 2.060
		Electrolytical	1.11	0.574– 2.180
Alternate Torsion	Fine	Mechanical		
		Endurance Limit = 14.3	19.5	1.48 –81.2
		Endurance Limit = 15.7	2.5	0.361– 4.16
		Electrolytical	15.4	2.43 –51.9

TABLE XIV.—VALUES OF "A" OBTAINED FROM APPLICATION OF HEYWOOD'S FORMULA TO AUTHOR'S TEST RESULTS.

Stress	Grain	Mode of Polishing	Average A, mm	Extreme Values of A
Alternate Tension-Compression	Fine	Mechanical	0.0287	0.0063–0.0394
		Electrolytical	0.0350	0.0063–0.0513
	Medium	Mechanical	0.0473	0.0137–0.0703
		Electrolytical	0.0462	0.0392–0.0599
	Coarse	Mechanical	0.0857	0.0508–0.1522
		Electrolytical	0.0528	0.0482–0.0572
Alternate Torsion	Fine	Mechanical		
		Endurance Limit = 14.3	0.0544	0.0222–0.0931
		Endurance Limit = 15.7	0.0372	0.0055–0.0685
		Electrolytical	0.0467	0.0220–0.1067

which permits calculation of the value of the "elementary structural unit A".

The average and extreme values of A obtained by application of the formula are given in Table XIII. This table shows that the difference between the limit values of A is very great and that A can hardly be considered as a constant. Therefore, we think that the formula of Neuber cannot be used. However, we must notice, with Kuhn and Hardrath, that the value of A calculated by the formula (2) is very sensitive to a small variation of q. Let us notice that, according to Kuhn and Hardrath (**11**), the value of A corresponding to $R_r = 57.1$ kg per sq mm ($= 81600$ psi) would be $A = 0.00623$ in. $=$ 0.158 mm. This value is not in agreement with the average values given heretofore.

The table below shows too that, with an equal tensile strength, the average value of A largely depends on the grain size of the metal.

2. *Heywood formula*

The Heywood formula is ((**4**), p. 351):

$$k_f = \frac{k_t}{1 + 2\sqrt{\frac{a}{r}}}$$

which may be written

$$a = \frac{r}{4}\left(\frac{k_t}{k_f} - 1\right)^2$$

Table XIV gives the average and extreme values of a by application of this formula to our test results. Here again, the differences between the limit values of a are very important.

It can be concluded from this examination that the laws of Neuber and of Heywood do not represent correctly enough the general behavior of variation of k_f with r.

APPENDIX II

Besides the hyperbolic notched specimens, fatigue resistance in tension-compression of flat specimens was studied. A transverse hole was bored in the specimen (Fig. 9) in order to compare with the results obtained by Roš (**13**), and to estimate the effect of the stress gradient upon the endurance limit.

These specimens have a width B. Holes having diameters which increase in geometrical progression following the series 0.5, 1, 2, 4, 8, 16, and 32 mm, are bored in the specimens.

The stress concentration factor is obtained within close approximation by the empirical formula

$$k_t = \frac{3\beta - 1}{\beta + 0.3} \left(\beta = \frac{B}{2r}\right) \ldots\ldots (1)$$

which is in agreement with the photo-elastic measurements of Hennig as well as with the theory of Howland.

The stress gradient can be estimated by the approximation method of Ruhl (**5**).

The distribution of the normal stresses on the section MN (Fig. 9) can be represented by the expression:

$$\sigma_x = \frac{\sigma_{\max}}{c'} \left(2 + c\,\frac{r^2}{x^2} + 3c\,\frac{r^4}{x^4}\right) \ldots (2)$$

which is a generalization of the formula of Kirsch

$$\sigma_x = \frac{\sigma_{\max}}{6} \left(2 + \frac{r^2}{x^2} + \frac{3r^4}{x^4}\right)$$

applicable to a plate of an infinite width.

The expression for the edge of the hole where $(x = r)$ reduces itself to $\sigma_{\max}$; then it follows from Eq 2 that:

$$c' = 2 + 4c \ldots\ldots\ldots\ldots (3)$$

Moreover, the average stress upon the width at MN must be equal to $\frac{\sigma_{\max}}{k_t}$; in order to fulfill this condition,

$$c = \frac{2(k_t - 1)}{4 - k_t D} \ldots\ldots\ldots\ldots (4)$$

where D represents the expression:

$$D = \frac{\beta^2(2\beta - 1) - 1}{\beta^3(\beta - 1)} \ldots\ldots\ldots (5)$$

The stress gradient at the point M is obtained by Eq (2):

$$G \equiv \left(\frac{d\sigma_x}{dx}\right)_{x=r} = \frac{14c\sigma_{\max}}{c'r} \ldots\ldots (6)$$

Thus the reduced gradient is:

$$\frac{rG}{\sigma_{\max}} = \frac{7c}{1 + 2c} = \frac{14(k_t - 1)}{k_t(4 - D)} \ldots\ldots (7)$$

By means of Eqs 1, 5 and 7, k_t and $\frac{rG}{\sigma_{\max}}$ can be calculated for all the specimens of the series; the results obtained are recorded in the Table XV (Fig. 14).

The value of $\frac{G}{\sigma_{\text{medium}}}$ is but little different from the value of $\frac{6.75}{r(\text{mm})}$ which is very close to the value $\frac{7}{r}$ corresponding to a hole drilled into a part of infinite width.

TABLE XV.—VALUES OF STRESS GRADIENTS FOR SERIES OF BORED SPECIMENS.

Series	Radius of Hole, r, mm	Width of Specimen, B, mm	$\beta = \frac{B}{2r}$	k_t	Reduced Gradient		Radius of Hyperbolic Notch Having Same Reduced Gradient, mm
					$\frac{rG}{\sigma_{\max}}$	$\frac{rG}{\sigma_{\text{medium}}}$	
No. 1............	0.25	45	90	2.98	2.34	6.96	0.358
No. 2............	0.5	45	45	2.96	2.34	6.92	0.567
No. 3............	1.0	50	25	2.925	2.35	6.87	0.910
No. 4............	2.0	60	15	2.88	2.36	6.80	1.48
No. 5............	4.0	64	8	2.77	2.41	6.67	2.35
No. 6............	8.0	64	4	2.56	2.50	6.40	3.64
No. 7............	16.0	70	2.187	2.24	2.79	6.26	....

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FATIGUE PROPERTIES OF COMPARABLE CAST AND WROUGHT STEELS*

By E. B. Evans,[1] L. J. Ebert[1] and C. W. Briggs[2]

Engineers and designers, in choosing between cast and wrought steels for use in the production of a particular part, must consider the physical and mechanical properties of both materials before an intelligent decision can be made. At present there is a lack of necessary information on the relative fatigue properties of cast and wrought steels. As a result, the Steel Founders' Society of America initiated a research program to supply this much needed data.

The work was divided into four main parts covering some of the more common factors which may influence the fatigue performance of standard-carbon and low-alloy steels, both cast and wrought, in the 0.30 to 0.40 per cent carbon range:

I. Effect of Steel Composition and Heat Treatment
II. Effect of Surface Finish
III. Directionality
IV. Section Size Effect (Mass Component)

These fatigue studies were carried out with small laboratory fatigue specimens tested in rotary bending, supplemented by tension and hardness tests for all steels, as well as by room-temperature impact tests for the cast steels used in the section size study. In Parts I and III both the notched and unnotched fatigue results are discussed.

MATERIAL, HEAT TREATMENT, AND PROCEDURE

The comparable cast and wrought steels used in this investigation included a cast and wrought 1040 carbon steel, four low-alloy cast steels (1330, 4135, 8630 and 8640) and four low-alloy wrought steels (1340, 4140, 4340 and 8640). In all, 19 different heats of steel (11 cast and 8 wrought) were tested. The cast steels were supplied in coupon form, the wrought steels in the form of hot-rolled stock. The identification and chemical analyses of the various heats are listed in Table I according to their use in the various phases of the investigation.

Prior to machining and testing, the different steels were heat treated in various ways. The plain-carbon series steels were either annealed or normalized and tempered, and the low-alloy steels were either normalized and tempered or quenched and tempered. The details of the heat treatments employed are given under each phase of the investigation. Except where otherwise indicated, all heat treating was done in the laboratory.

Fatigue tests were conducted on standard R. R. Moore high-speed (10,000 rpm) rotating-beam machines. The unnotched and notched specimens employed are shown in Fig. 1.

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TABLE I.—IDENTIFICATION AND CHEMICAL ANALYSIS OF THE CAST AND WROUGHT STEELS TESTED IN THE VARIOUS PHASES OF THE INVESTIGATION.

	Steel	Chemical Composition, per cent								Remarks
		Carbon	Manganese	Silicon	Phosphorus	Sulfur	Chromium	Nickel	Molybdenum	
PART I: EFFECT OF STEEL COMPOSITION AND HEAT TREATMENT										
Nos. 1 and 3........	Cast 1040	0.39	0.78	0.47	0.023	0.039	...	...	...	1 in. Coupons
Nos. 2 and 4........	Wrought 1040	0.39	0.82	0.28	0.020	0.037	...	...	...	Hot-Rolled ¾-in. Round
Nos. 5 and 14........	Cast 1330	0.31	1.64	0.47	0.015	0.022	...	...	...	1-in. Coupons
No. 6..............	Cast 1330	0.30	1.50	0.42	0.031	0.043	...	...	...	1-in. Coupons
Nos. 7 and 15........	Wrought 1340	0.38	1.68	0.35	0.020	0.025	...	...	...	Hot-Rolled ¾-in. Round
Nos. 8 and 16........	Cast 4135	0.37	0.82	0.45	0.035	0.027	0.88	...	0.19	1-in. Coupons
Nos. 9 and 17........	Wrought 4140	0.39	0.84	0.28	0.019	0.029	0.96	...	0.19	Hot-Rolled ¾-in. Round
Nos. 10 and 18........	Cast 4335	0.34	0.60	0.49	0.034	0.036	0.70	1.74	0.30	1-in. Coupons
Nos. 11 and 19........	Wrought 4340	0.39	0.76	0.30	0.035	0.013	0.78	1.79	0.25	Hot-Rolled ¾-in. Round
Nos. 12 and 20........	Cast 8630	0.29	0.73	0.45	0.026	0.035	0.52	0.57	0.23	1-in. Coupons
Nos. 13 and 21........	Wrought 8640	0.42	0.89	0.30	0.040	0.014	0.57	0.61	0.22	Hot-Rolled ¾-in. Round
PART II: EFFECT OF SURFACE FINISH										
No. 22..............	Cast 8640	0.44	1.00	0.36	0.014	0.027	0.60	0.58	0.23	1-in. Coupons
No. 23..............	Wrought 8640	0.42	0.89	0.30	0.040	0.014	0.57	0.61	0.22	Hot-Rolled ¾-in. Round
No. 24..............	Cast 8640	0.41	0.94	0.35	0.014	0.027	0.68	0.59	0.22	Cast-to-shape Fatigue Specimens with Riser
No. 25..............	Cast 8640	0.40	0.95	0.37	0.015	0.027	0.60	0.58	0.23	Same as Steel 24 Without Risers
PART III: DIRECTIONALITY										
No. 26..............	Wrought E-4140	0.40	0.88	0.33	0.017	0.033	1.00	...	0.017	Hot-Rolled 4-in. Round
PART IV: SECTION SIZE EFFECT										
No. 27..............	Cast 1030	0.32	0.77	0.47	0.042	0.038	...	...	...	1¼-in., 3-in. and 6-in. Coupons
No. 28..............	Wrought C-1030	0.33	0.83	...	0.015	0.034	...	...	...	Hot-Rolled 6¼-in. Square Bloom
Nos. 29 and 30........	Cast 8635	0.36	0.89	0.50	0.049	0.040	0.59	0.38	0.32	1¼-in., 3-in. and 6-in. Coupons

Except for the surface finish study, all unnotched specimens were carefully ground and lapped longitudinally to remove all traces of circumferential scratches before testing. The notched specimens were polished to a high finish at the base of the notch, employing a 0.030-in. diameter wire lap and a fine abrasive.

Eight to eleven fatigue specimens were tested to establish each *S-N* (applied stress *versus* number of cycles to failure) curve. The endurance limit was taken as the maximum stress borne for 10 million reversals without failure. Unless otherwise pointed out, the curves were obtained as "lines of best fit" with the exception that the endurance limit was based on test results which showed no failure after 10 million cycles and which was always less than the individual test results of specimens which had failed.

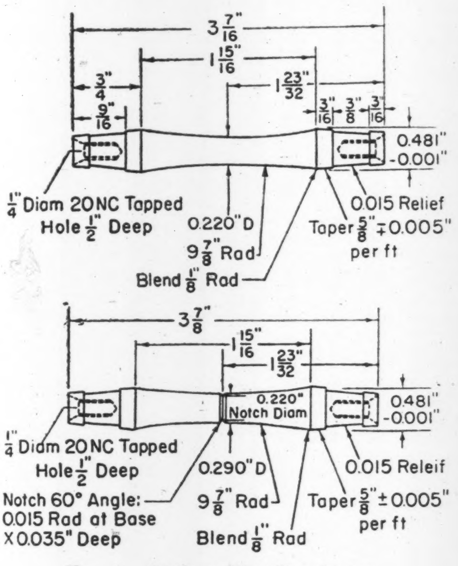

Fig. 1.—Fatigue Test Specimens.

In conjunction with each *S-N* curve, standard ½-in. diameter tension specimens were tested at least in duplicate. The broken tension test specimens served as sections for standard Brinell hardness tests. Standard Charpy V-notch impact tests at room temperature were performed, at least in duplicate, in conjunction with each *S-N* curve determined for the cast steels in the section size study.

Part I.—Effect of Steel Composition and Heat Treatment

The studies in this phase of the investigation included a plain-carbon steel and four low-alloy steels which were studied in the unnotched and notched condition. The five cast steel grades were 1040, 1330, 4135, 4335, and 8630; the five comparable wrought steels were 1040, 1340, 4140, 4340, and 8640. The plain-carbon steels were tested in the full annealed and in the normalized-and-tempered condition; the four low-alloy steels were tested in the normalized-and-tempered and in the quenched-and-tempered conditions. For each steel series for a given heat treatment the tensile strength level of the cast and the wrought steel was the same. In all, the five steel series encompassed a tensile strength range of 80,000 to 170,000 psi.

The identification, heat treatment, tensile properties, hardness, and fatigue test results are listed in Table II. Typical fatigue curves (*S-N* curves) are shown in Figs. 2 to 5 for the unnotched and notched conditions.

Unnotched Fatigue Properties:

The *S-N* curves for all the unnotched wrought steels showed but little scatter in the test data for all the steels investigated. Although the data for the comparable cast steels showed somewhat more scatter, the general shape of each curve and the endurance limit for 10 million cycles appeared to be well defined. The *S-N* curves for the wrought steels all exhibited a sharp knee at about 10^6 cycles; all the cast steels seemingly exhibited a relatively continuous curve.

TABLE II.—TENSILE, HARDNESS, AND FATIGUE TEST RESULTS FOR STEELS USED IN PART I.

	Steel	Heat Treatment,[a] deg Fahr	Tensile Strength, psi	Yield Strength, 0.2 per cent offset, psi	Reduction of Area, per cent	Elongation in 2 in., per cent	Brinell Hardness Number	Endurance Limit, psi		Endurance Ratio	
								Unnotched	Notched	Unnotched	Notched
				Annealed[a]							
No. 1	Cast 1040	1650	83 500	49 300	46.7	27.5	156	33 200	26 000	0.40	0.31
No. 2	Wrought 1040	1650	81 400	47 600	54.6	31.0	149	39 300	25 800	0.48	0.32
				NORMALIZED AND TEMPERED[b]							
No. 3	Cast 1040	N1650, T1200	94 200	56 000	52.2	24.5	187	37 700	28 000	0.40	0.30
No. 4	Wrought 1040	N1650, T1200	90 000	55 700	58.3	26.5	170	43 500	27 200	0.48	0.30
No. 5	Cast 1330	N1650, T1200	99 300	61 500	58.5	24.0	201	48 400	31 700	0.49	0.32
No. 6	Cast 1330	N1650, T800	97 000	63 500	58.5	26.0	201	41 700	31 200	0.43	0.32
No. 7	Wrought 1340	N1650, T1200	101 800	56 800	60.1	23.5	207	58 700	33 000	0.58	0.32
No. 8	Cast 4135	N1650, T1200	112 700	86 500	43.1	18.0	223	51 200	33 300	0.45	0.30
No. 9	Wrought 4140	N1650, T1200	111 100	86 100	62.9	23.4	223	62 000	31 200	0.56	0.28
No. 10	Cast 4335	N1650, T1125	126 500	100 500	50.2	14.5	262	63 000	34 900	0.50	0.28
No. 11	Wrought 4340	N1650, T1200	124 600	98 900	63.8	23.5	262	75 500	35 000	0.60	0.28
No. 12	Cast 8630	N1650, T1200	110 500	85 600	53.7	19.0	223	54 000	33 100	0.49	0.30
No. 13	Wrought 8640	N1650, T1200	108 500	82 800	60.2	24.0	217	64 500	32 000	0.59	0.30
				QUENCHED AND TEMPERED[b, c]							
No. 14	Cast 1330	Q1550, T1150	122 200	106 000	55.6	20.5	269	58 500	37 300	0.48	0.31
No. 15	Wrought 1340	Q1525, T1200	121 200	106 900	60.9	22.5	269	68 400	36 300	0.56	0.30
No. 16	Cast 4135	Q1550, T1175	146 400	131 000	35.8	14.0	311	61 300	40 600	0.42	0.28
No. 17	Wrought 4140	Q1525, T1150	146 800	133 000	60.1	18.5	311	87 400	41 200	0.59	0.28
No. 18	Cast 4335	Q1525, T1025	168 200	155 900	17.8	9.5	375	77 600	48 200	0.46	0.29
No. 19	Wrought 4340	Q1500, T1025	168 400	158 100	56.3	17.0	375	96 500	45 900	0.57	0.27
No. 20	Cast 8630[d]	Q1550, T1200	137 500	125 800	34.5	14.8	286	64 900	38 600	0.47	0.28
No. 21	Wrought 8640	Q1525, T1200	138 200	124 200	65.6	21.5	286	77 800	37 400	0.55	0.27

[a] N = Normalized. T = Tempered. Q = Quenched.

[b] All test specimens were taken from the approximate center of the original material.

[c] Slightly oversize test specimens were first machined from the original material in the normalized and tempered condition. These specimens were then water quenched and tempered, and ground-to-size. Tempering was followed by a water quench.

[d] Tensile test results based on 0.22 in. diam and 0.88 in. gage length.

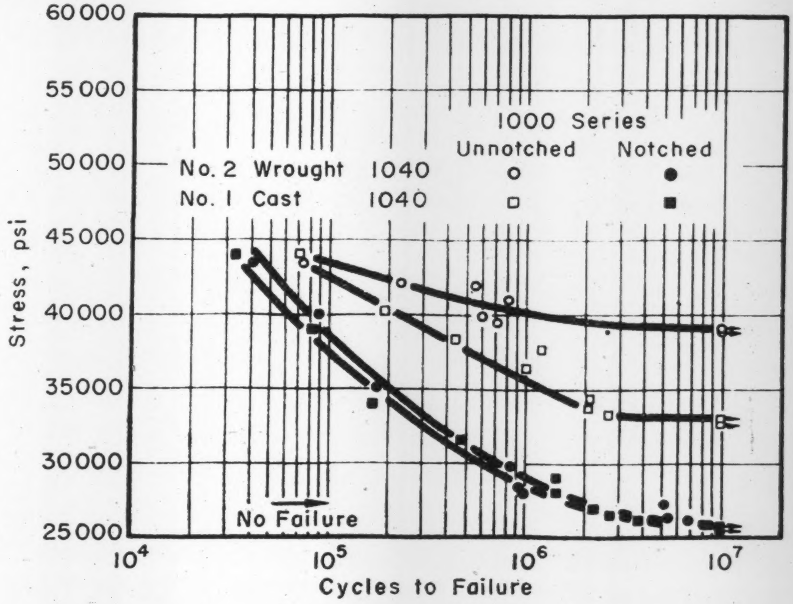

FIG. 2.—*S-N* Curves for Cast 1040 and Wrought 1040 Steel (Notched and Unnotched) Annealed Condition.

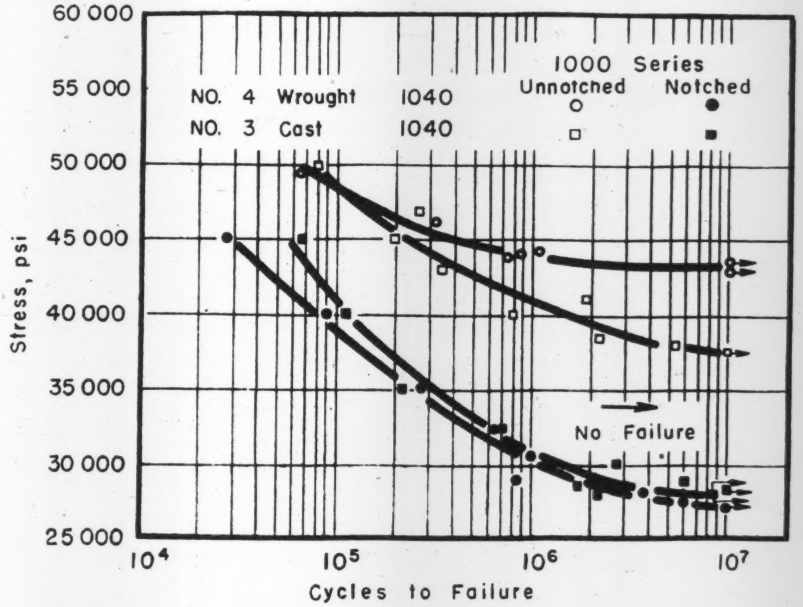

FIG. 3.—*S-N* Curves for Cast and Wrought 1040 Steels (Notched and Unnotched) Normalized and Tempered to Same Strength Level.

In addition, each wrought steel had a flatter type *S-N* curve than the comparable cast steel.

ment when necessary, it was found in all cases that the endurance limit of the cast steel was about $\frac{4}{5}$ the longitudinal endurance limit of the comparable wrought steel. This condition is shown in Fig. 6. This relationship appeared to be unaffected by steel composition and type of heat treatment.

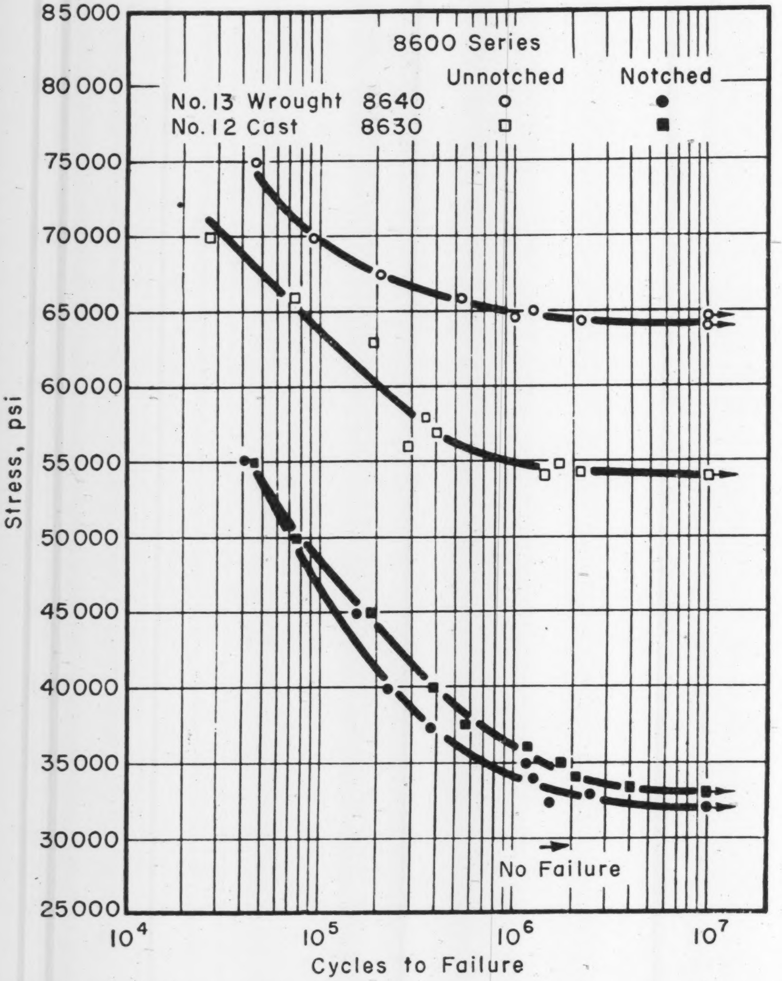

FIG. 4.—*S-N* Curves for Cast and Wrought 8600 Series Steel (Notched and Unnotched) Normalized and Tempered to Same Strength Level.

A direct comparison of the endurance limit of cast and wrought steels is difficult on a quantitative basis unless both have the same tensile strength. Since the tensile strengths were not always equal, it was necessary to adjust the cast endurance limit. By applying this adjust-

The endurance ratio for plain-carbon steels in both the fully annealed and in the normalized-and-tempered conditions was 0.40 for the cast steel and 0.48 for the wrought steel. The endurance ratio for the four alloy series steels in both the normalized-and-tempered and in the quenched-and-tempered conditions encompassed a range of 0.42 to 0.50 for the cast steels and for the wrought steels a range of 0.55 to 0.60. These results show that for a given material—cast or wrought—the low-alloy steels exhibit somewhat higher values of endurance ratio than those of the plain-carbon steels.

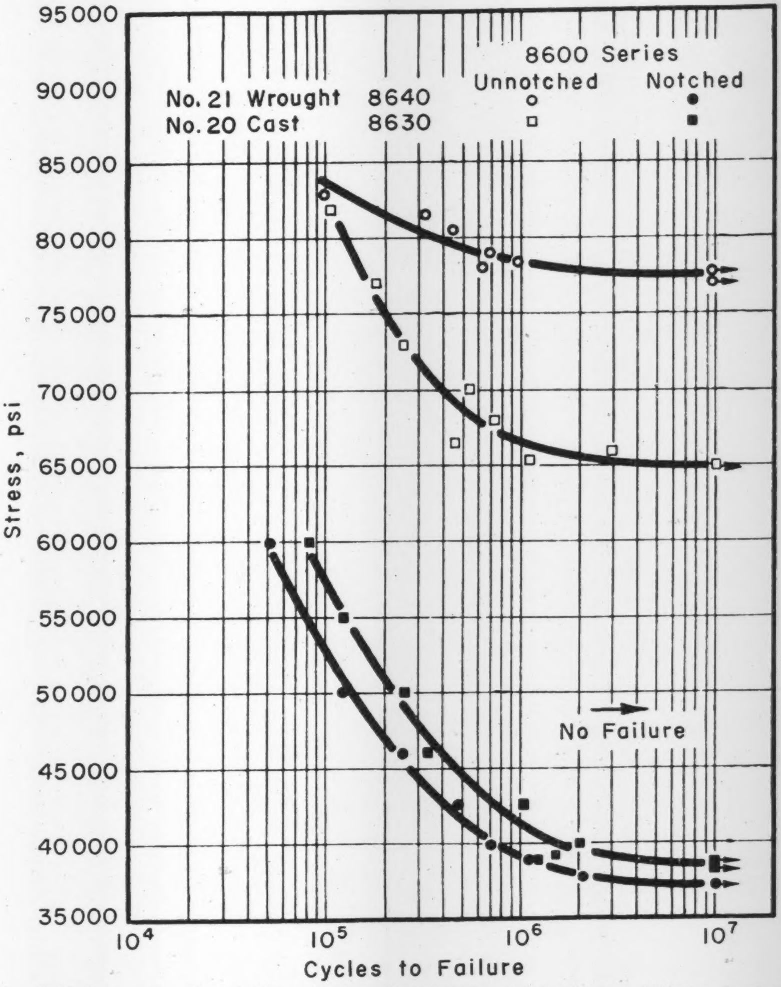

FIG. 5.—*S-N* Curves for Cast 8630 and Wrought 8640 Steel (Notched and Unnotched) Hardened and Tempered to the same Strength Level.

Notched Fatigue Properties:

External notches of one sort or another are nearly always present in commercial steel components because of design requirement, machining methods or discontinuities; therefore, the notch effects should be considered for both cast and wrought materials. In this

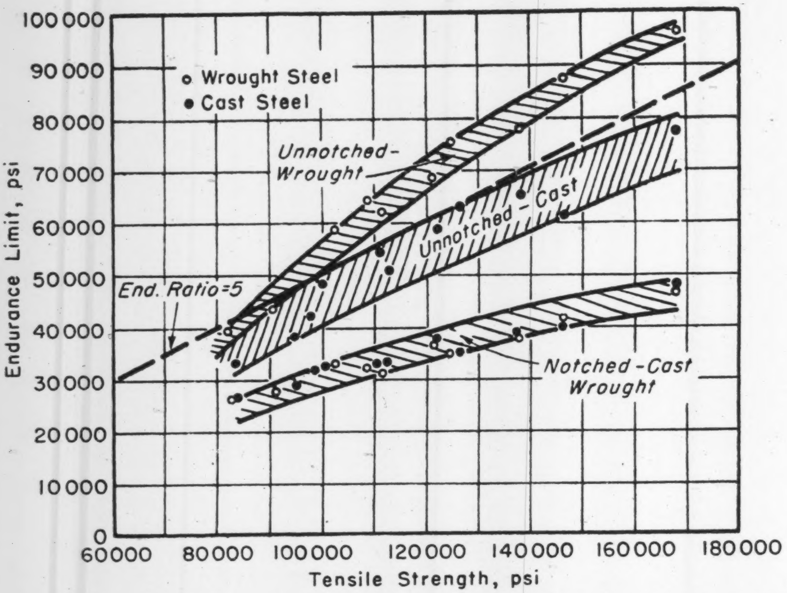

FIG. 6.—Variation of Endurance Limit with Tensile Strength for Comparable Cast and Wrought Steels.

TABLE III.—FATIGUE STRENGTH REDUCTION AND NOTCH SENSITIVITY FACTORS FOR COMPARABLE CAST AND WROUGHT STEELS.

For $N = 10^7$ cycles and $K_t = 2.2$

	Steel	Tensile Strength, psi	Fatigue Strength Reduction Factor, K_a	Notch Sensitivity, q
		Annealed		
No. 1	Cast 1040	83 500	1.28	0.23
No. 2	Wrought 1040	81 400	1.52	0.43
		Normalized and Tempered		
No. 3	Cast 1040	94 200	1.35	0.29
No. 4	Wrought 1040	90 000	1.60	0.50
No. 5	Cast 1330	99 300	1.53	0.44
No. 6	Cast 1330	97 000	1.34	0.28
No. 7	Wrought 1340	101 800	1.78	0.65
No. 8	Cast 4135	112 700	1.54	0.45
No. 9	Wrought 4140	111 100	1.99	0.81
No. 10	Cast 4335	126 500	1.81	0.68
No. 11	Wrought 4340	124 600	2.16	0.97
No. 12	Cast 8630	110 500	1.63	0.53
No. 13	Wrought 8640	108 500	2.02	0.85
		Quenched and Tempered		
No. 14	Cast 1330	122 200	1.57	0.48
No. 15	Wrought 1340	121 200	1.88	0.73
No. 16	Cast 4135	146 400	1.51	0.43
No. 17	Wrought 4140	146 800	2.12	0.93
No. 18	Cast 4335	168 200	1.61	0.51
No. 19	Wrought 4340	168 400	2.10	0.92
No. 20	Cast 8630	137 500	1.68	0.57
No. 21	Wrought 8640	138 200	2.08	0.90

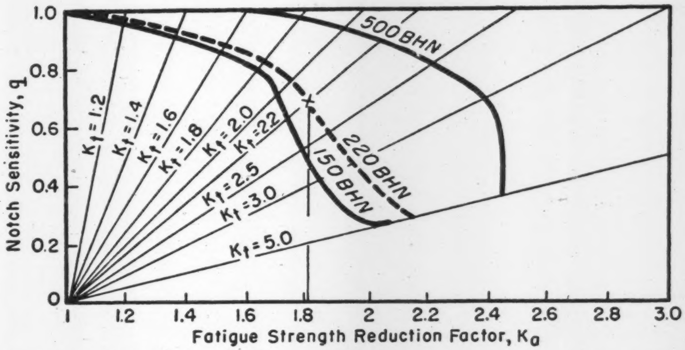

FIG. 7.—Notch Sensitivity, q, Theoretical Stress Concentration Factor, K_t, and Fatigue Strength Reduction Factor, K_a, for Wrought Steel at Various Hardness Levels. (Johnson and Lipson (2).)

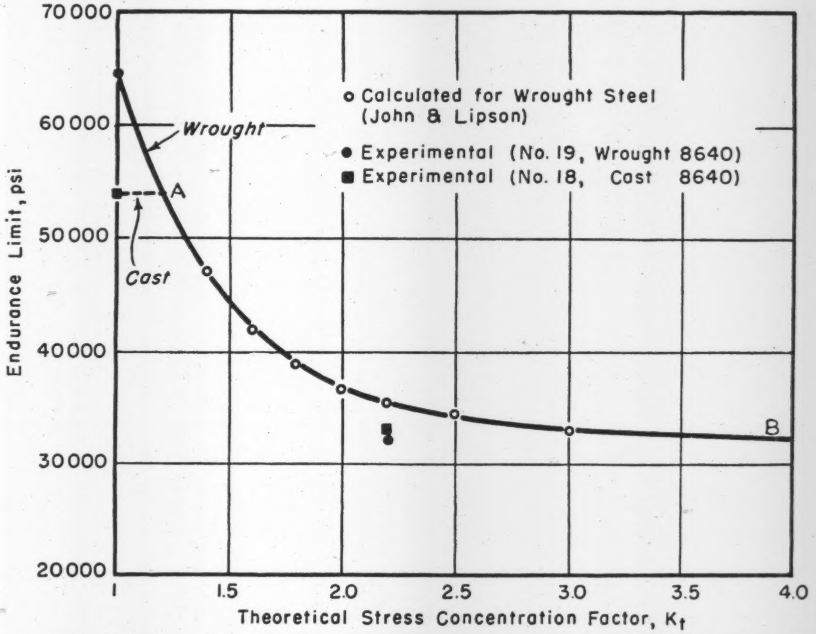

FIG. 8.—Effect of Increasing Stress Concentration on the Endurance Limit of Steel at a Hardness Level of BHN 220 or a Tensile Strength Level of 110,000 psi. In the Region From A to B the Curves for Cast and Wrought Material Coincide.

phase of the investigation, notched fatigue curves were established for the same materials tested in the unnotched condition. The notched fatigue curves for both cast and wrought material (Figs. 3 to 5) exhibited the same general shape of curve, that is, a continuously decreasing stress with increasing number of cycles to failure and with no sharp discontinuities evident. The data indicated very little scatter in either cast or wrought steels.

In comparing each steel series for a given heat treatment, it is evident that the endurance limit of the notched cast steel is about the same as that of the comparable wrought steel—in fact, the fatigue curves are nearly superimposed upon one another. The plain-carbon and the four low-alloy series steels heat treated various ways encompassed an endurance ratio range of 0.28 to 0.32 for the cast steels and 0.27 to 0.32 for the wrought steels. Therefore, there is no advantage of one material over the other at either small or large numbers of cycles when critically shaped notches are present in steels tested in fatigue.

The cast and wrought materials can also be compared as to their notch sensitivity, q. The notch sensitivity is a factor used to account for the dimensionless difference between the theoretical stress concentration factor and the actual factor obtained in fatigue tests, and is usually defined as:

$$q = \frac{K_a - 1}{K_t - 1}$$

where:

K_a = $\frac{\text{unnotched endurance limit}}{\text{notched endurance limit}}$ = fatigue strength reduction factor, and

K_t = theoretical stress concentration factor = 2.2 for this investigation (calculated from paper by Peterson (**1**)).

If $q = 0$, the material is not notch sensitive. If $q = 1$, the full theoretical notch effect is attained.

TABLE IV.—TENSILE, HARDNESS, AND FATIGUE TEST RESULTS FOR STEELS USED IN STUDY OF EFFECT OF SURFACE FINISH.

	Steel	Tensile Strength, psi	Yield Strength, 0.2 per cent Offset, psi	Reduction of Area, per cent	Elongation in 2 in., per cent	Brinell Hardness Number	Surface Finish	Endurance Limit, psi	Endurance Ratio
No. 22.......	Cast 8640	112 100	82 400	37.0	19.0	223	Polished	50 000	0.45
							Lathe-Turned	50 000	0.45
No. 23.......	Wrought 8640	108 500	82 700	60.2	24.0	217	Polished	64 500	0.59
							Lathe-Turned	50 000	0.46
No. 24.......	Cast 8640[a]	109 000	...	...	...	217	As-Cast (Risered)	35 000	0.32
No. 25.......	Cast 8640[a]	108 500	...	...	...	217	As-Cast (Not Risered)	35 000	0.32

[a] For the two heats of cast-to-shape fatigue specimens, the tensile strengths were determined with small ¼ in. diameter test specimens; other results are based on standard 0.505 in. diameter test specimens.

NOTE.—All steels normalized at 1650 F for 1 hr and tempered at 1200 F for 1 hr.

The values of q as shown in Table III show that the cast steels are less notch sensitive (or more notch resistant) than the comparable wrought steels at all tensile strength levels. In addition, the notch sensitivity of both materials increases as the tensile strength level increases, that is, the higher strength steels are more affected by a notch than are the softer steels, as evident in Fig. 6.

Although the notch dimensions were held constant during this investigation, the influence of varying notch severity on the endurance limit may be predicted from the curves by Johnson and Lipson (**2**)[3] reproduced in Fig. 7. For example, referring to wrought 8640 steel (No. 13), for a theoretical stress concentration of 2.2 and a hardness level of BHN 220 (or tensile strength level of 110,000 psi) a fatigue strength reduction factor of 1.82 is obtained (Fig. 7). The notched endurance limit is then readily calculated as 35,400 psi since the strength reduction factor is the ratio of the unnotched endurance limit to the notched endurance

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper see p. 1010.

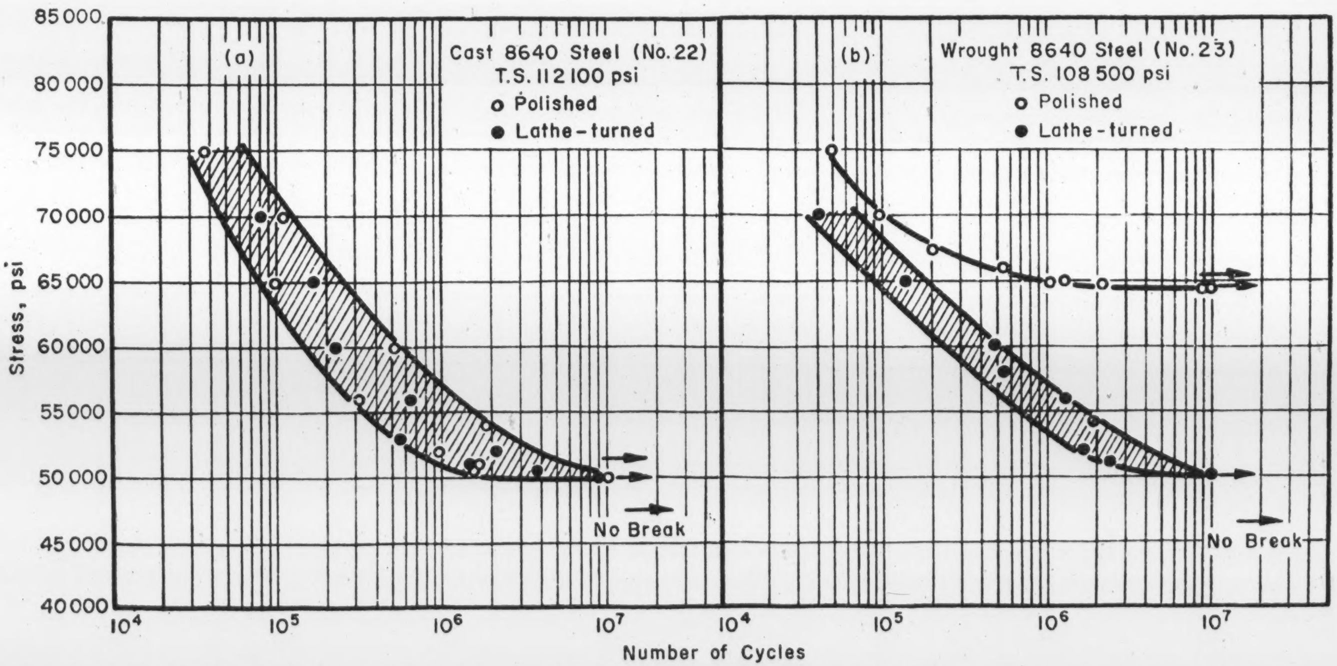

FIG. 9.—Fatigue Test Results for a Cast and a Wrought 8640 Steel with a Polished and a Lathe-Turned Finish. Normalized and Tempered to the Same Strength Level.

limit and the unnotched endurance limit is known (64,500 psi).

The calculated values for this steel are shown in Fig. 8, revealing a smooth decrease of the endurance limit with increasing theoretical stress concentration. In contrast, the expected variation of endurance limit with increasing notch severity for the comparable cast steel (No. 12) is drawn so that a plateau exists at mild stress concentrations meeting the curve for wrought material at point *A*. The existence of the plateau for cast steels, that is, the very mild external notches have no effect on the endurance limit, seems justified in view of the following.

The endurance limit of a cast steel with a lathe-turned finish was the same as one with a polished finish, whereas, a lathe-turned finish for wrought steel reduced the endurance limit to a value corresponding to that of the cast steel. (The effect of surface on the fatigue properties is discussed in Part II.) Thus, it seems reasonable to expect that the endurance limit of the cast and the wrought steel will become equal at some point, say, point *A* in Fig. 8, if a machined surface is considered as a mild stress concentration. From point *A* to *B*, the calculated curves for both cast and wrought materials were drawn so that they coincided, because the experimentally determined endurance limit for a theoretical stress concentration of 2.2 was essentially the same for comparable cast and wrought steels, and these points fall close to the calculated curve. Fair agreement between the experimentally determined points and the calculated points can be shown for all steels tested in this investigation.

PART II.—SURFACE FINISH

Steel components subjected to fatigue service seldom meet the surface conditions of polished laboratory test specimens. The fatigue properties of wrought steel are known to decrease substantially as the surfaces vary from ground, to lathe-turned, to hot-rolled, and to as-forged. Thus in comparing cast and wrought steels, the design engineer must consider the probable extent of damage with different surface finish for each material.

The studies of Part II cover the unnotched fatigue properties of (*a*) a cast 8640 steel with a polished, a lathe-turned, and an as-cast finish; and (*b*) a wrought 8640 steel with a polished and a lathe-turned finish. Both the cast and the wrought steel were normalized and tempered to a tensile strength level of 110,000 psi. The specimens with an as-cast finish were cast-to-shape with and without risers in an attempt to determine any effect due to centerline shrinkage.

The identification, tensile properties, hardness, and fatigue properties are given in Table IV.

Polished and Lathe-Turned Finish (Cast versus Wrought):

The fatigue data for a polished and a lathe-turned finished cast 8640 steel are plotted in Fig. 9(*a*). A band has been drawn encompassing the test results for the polished finish. The results with a lathe-turned finish all fell within the lower two-thirds of this band, thus indicating that a lathe-turned surface has very little effect in lowering the fatigue properties. In fact, the endurance limit for both finishes was the same—50,000 psi, or an endurance ratio of 0.45 for both finishes.

The results for the comparable finishes for the wrought steel, Fig. 9(*b*), shows that the lathe-turned finish exerted an appreciable damaging influence on the fatigue life, and was more pronounced at the lower stress levels. The endurance limit was lowered from

64,500 psi for the polished finish to 50,000 psi for the lathe-turned finish corresponding to a decrease in the endurance ratio from 0.59 to 0.46. This amounts to about 28 per cent reduction in endurance limit.

A comparison of the fatigue properties of the cast and wrought 8640 steel showed that for a polished finish the endurance limit of the cast steel was about $\frac{4}{5}$ that of the wrought steel, and for a lathe-turned finish, the fatigue properties of the cast and wrought 8640 steel are very nearly equal.

As-Cast Finish (With and Without Centerline Shrinkage):

The fatigue test results for cast-to-shape specimens made with and without risers and tested with an as-cast finish are shown in Fig. 10. A band has been drawn enclosing the test results for those specimens which were risered. The results with specimens made without risers all fall within this band, thus indicating that the degree of centerline shrinkage present[4] had no damaging influence on the fatigue properties. In both cases, the endurance limit was 35,000 psi or an endurance ratio of 0.32. It is not surprising that centerline shrinkage appeared to exert no effect on the fatigue properties. In bending fatigue, the stress in any fiber of a specimen is proportional to the distance from the neutral axis; therefore defects near the neutral axis are less likely to result in early fatigue failure than those defects at or near the surface.

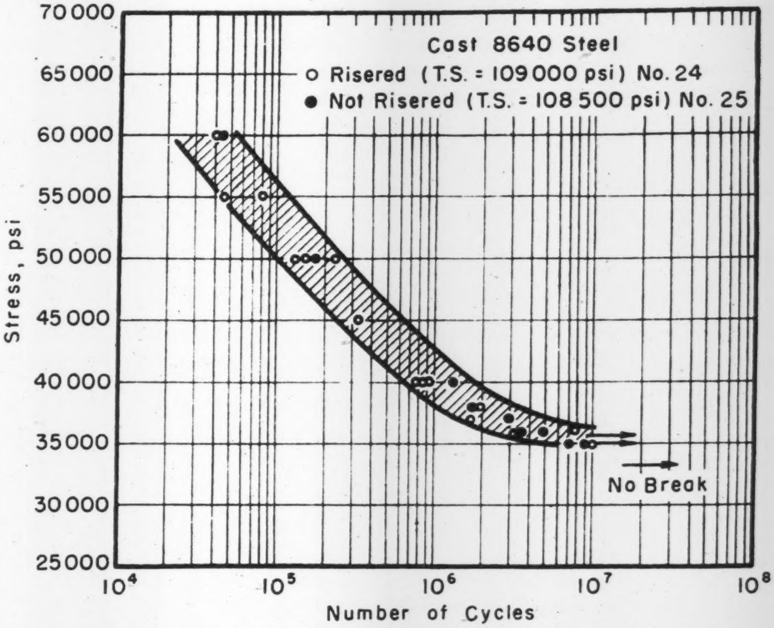

FIG. 10.—Fatigue Test Results for Cast 8640 Steel With an As-Cast Finish. Specimens Cast-to-Shape with and Without Risers, Normalized and Tempered to the Same Strength Level.

When the endurance limit for polished and as-cast finishes are compared, it is noted that the as-cast finish lowered the endurance limit about 30 per cent. A comparable finish for a wrought steel, for example, a hot-rolled or as-forged finish, is known to decrease the endurance limit about 40 per cent at a tensile strength level of 110,000 psi (2). These comparisons indicate the extent of damage to be expected with the worst surface finish for cast and wrought steel, respectively.

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[4] The microstructure at the center of an unrisered specimen indicated that the extent of centerline shrinkage was slight.

It should be mentioned that there are two other factors in addition to the as-cast finish which may have contributed to the decrease in fatigue properties:

A. *Surface Decarburization.* An examination of the microstructure at the surface revealed slight decarburization (about 0.002 in.), even though the heat treatment was carried out in a neutral salt bath. The injurious effect of a decarburized skin on the fatigue strength is well known; however, it is not possible to determine the contribution of this effect in the presence of the detrimental influence of the surface irregularities associated with the as-cast finish.

TABLE V.—TENSILE, HARDNESS, AND FATIGUE TEST RESULTS FOR WROUGHT 4140 STEEL (NO. 26) USED IN DIRECTIONALITY STUDY.

Direction of Test	Tensile Strength, psi	Yield Strength, 0.2 per cent Offset, psi	Reduction of Area, per cent	Elongation in 2 in., per cent	Brinell Hardness Number	Endurance Limit, psi		Endurance Ratio	
						Unnotched	Notched	Unnotched	Notched
Transverse..........	110 700	81 000	29.5	13.0	217	53 000	26 000	0.48	0.24
Longitudinal........	110 100	80 400	60.7	24.5	217	61 000	28 000	0.55	0.25

B. *Inertia Stresses.* The specimens were cast slightly out-of-round, which, in conjunction with the slight eccentricity resulting from machining, may have set up sufficiently high inertia stresses so as to lead to premature failure.

In view of these considerations, it is not clear to what extent the as-cast finish, in itself, decreased the fatigue properties.

PART III.—DIRECTIONALITY

Another factor to be considered in the design of steel parts for fatigue service is the directionality or anisotropic effect for wrought steel, an effect not inherent with cast steel. The directionality effect has been studied by many investigators with tensile and notched-bar impact tests. It has been found that directionality has little effect on tensile strength but is reflected by a loss of ductility in the tension test and also by a loss of toughness in the impact test.

There are relatively few published data on the effect of directionality in fatigue, and until recently no serious attempt appears to have been made to study this effect in fatigue. Ransom and Mehl (3) determined the fatigue properties of SAE 4340 steel forgings in the longitudinal (direction of working) and transverse directions. For both low- and high-quality steel (quality measured by transverse reduction of area) the tensile strength level of 125,000 psi was the same in both directions. Anisotropy was found to be very marked and was greater for poorer quality steel. For high quality steel the average transverse endurance limit was about 84 per cent of the average longitudinal endurance limit, but for low-quality steel it was only 68 per cent of the longitudinal value.

The study of directionality carried out in this investigation covered the longitudinal and transverse fatigue properties, notched and unnotched, of specimens from the center of a 4-in. hot-rolled round of E-4140 steel. The steel was normalized and tempered to a strength level of 110,000 psi, which was comparable to the cast 4135 steel (No. 8).

The tension and fatigue test results are summarized in Table V and the *S-N* curves are shown in Fig. 11.

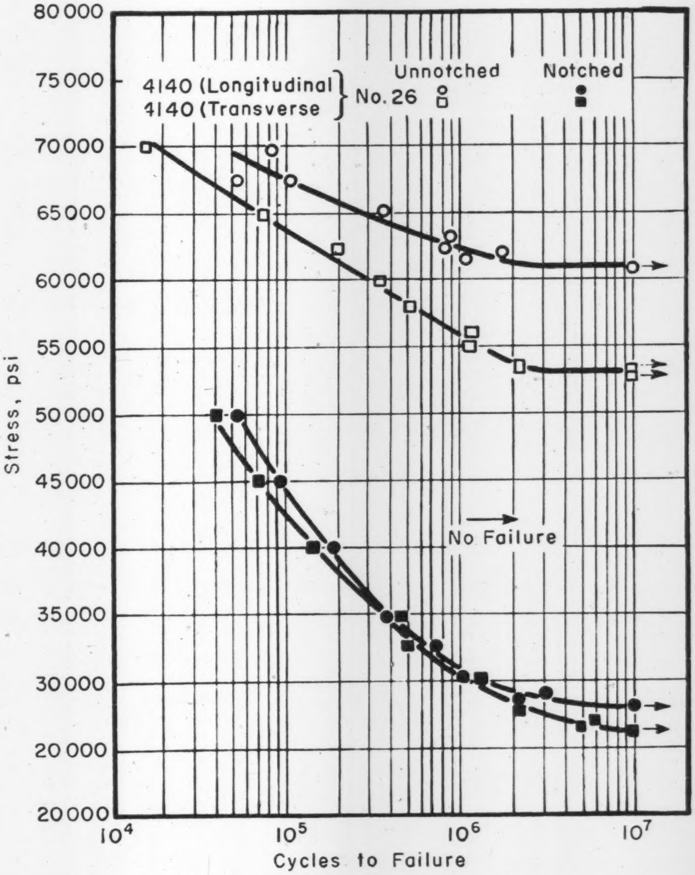

FIG. 11.—*S-N* Curves for a Wrought 4140 Steel (Notched and Unnotched) in the Longitudinal and Transverse Direction: Normalized and Tempered.

Unnotched Fatigue Tests:

The unnotched fatigue curves for both the longitudinal and transverse directions showed little scatter in the test data. Both curves exhibited a sharp knee at about 2 million cycles; however, the longitudinal direction had a flatter type *S-N* curve. The anisotropic effect of the wrought steel was indicated by the lower transverse endurance limit (53,000 psi), which was 87 per cent of the longitudinal endurance limit (61,000 psi).

This large directionality effect cannot be correlated with tensile or yield strength because these properties were independent of direction, that is, the tensile as well as the yield strength was the same in the longitudinal and transverse directions. However, the tensile ductility (reduction of area and elongation) was dependent upon direction.

In this investigation the ductility in the transverse direction was only one-half that in the longitudinal direction.

Presumably, then, the directionality effect in the tensile ductility is reflected in the unnotched fatigue properties. However, an exact correlation is hardly possible without a detailed study of such factors as melting practice and reduction ratio in working and how they affect amount, size, shape, and distribution of inclusions. For example, in this study the low transverse reduction of area in the tension test indicates a poor quality steel, yet the transverse endurance limit was 87 per cent of the longitudinal endurance limit which, according to Ransom and Mehl, is about the same value obtained for a high-quality steel.

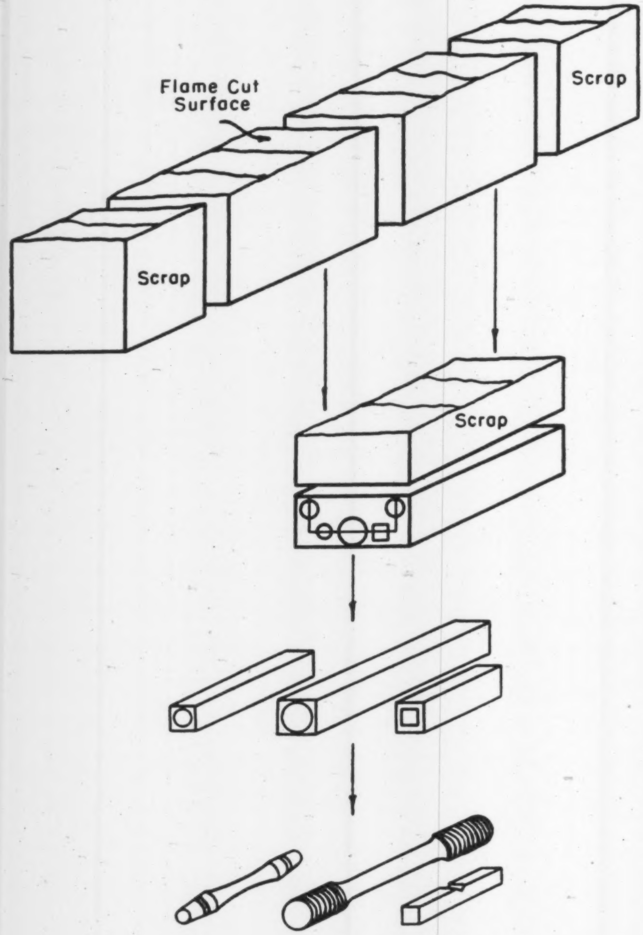

FIG. 12.—Method of Obtaining Test Specimens from Cast Coupon. (Location Shown Is $\frac{3}{8}$ in from Surface of 3 in. Section.)

It is interesting to note that the longitudinal fatigue properties of specimens from the $\frac{3}{4}$-in. round wrought 4140 steel (see Table II, Steel No. 9) were about the same as the longitudinal properties of specimens from the center of the

4-in. round. Although two different heats of steel are under consideration, it would appear that the size of the wrought section from which samples are taken has little effect on the unnotched fatigue properties, within the limits studied here

A comparison of the results of this study with those previously obtained for a cast 4137 steel at the same strength level (No. 8) reveals that the endurance ratio for the cast steel (0.45) is lower than the endurance ratio of the wrought steel in the longitudinal direction (0.55), but about the same as the wrought steel in the transverse direction (0.48). But, as Ransom and Mehl have reported, the transverse endurance ratio for wrought steel can be as low as 0.37 for poor-quality steel. In any case, these comparisons serve to point out that the directionality effect in unnotched fatigue is very real and can be most severe. Moreover, it should be considered when rating the relative merits of cast and wrought steels.

Notched Fatigue Tests:

The longitudinal and transverse notched fatigue curves exhibit the same general shape, that is, a continuously increasing number of cycles to failure with decreasing stress and with no sharp discontinuities evident. The data indicate very little scatter for both directions.

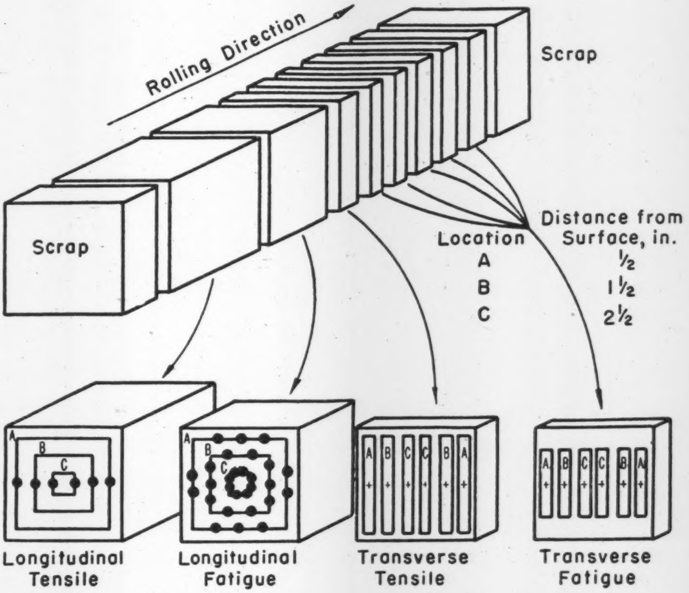

FIG. 13.—Method of Obtaining Test Specimens from Various Locations in the Longitudinal and Transverse Directions of a 6¼ in. Square Section of a C1030 Bloom.

The notched endurance ratio in the transverse direction (0.24) was slightly lower than the notched endurance ratio in the longitudinal direction (0.25), the difference in ratio corresponding to a slight advantage of 2000 psi in notched endurance limit for the longitudinal direction. Thus, the directionality effect is not readily apparent with externally notched specimens of wrought steel.

The notched endurance ratio of cast 4137 steel (0.30) and wrought ¾-in. 4140 steel (0.28) showed superior notched properties over the 4-in. wrought 4140 steel considered in either direction. The lower notched properties for specimens from the 4-in. section of wrought steel may be an indication that the size of a wrought section from which specimens are taken has some effect on the notched properties. As noted above, this mass effect was not apparent with unnotched specimens.

TABLE VI.—SECTION SIZE EFFECT DATA ON TENSILE, HARDNESS, AND IMPACT PROPERTIES OF CAST 1030 STEEL NO. 27 NORMALIZED FROM 1600 F AND TEMPERED AT 1200 F.

Position Code Letter	Distance from Surface, in.	Tensile Strength, psi	Yield Point,[a] psi	Reduction of Area, per cent	Elongation in 2 in., per cent	Brinell Hardness Number	Impact Energy,[b] ft-lb
			1¼-IN. SECTION SIZE				
A.	⅝	81 600	44 800	48.5	29.5	156	20
B.	5/16	82 200	45 900	52.8	30	163	19.5
			3-IN. SECTION SIZE				
C.	1 1/16	78 400	40 800	45.3	29	156	12.5
D.	¾	78 500	40 800	47.0	29.5	159	11.5
E.	⅜	79 500	42 000	49.6	30	156	14
			6-IN. SECTION SIZE				
F.	2½	76 400	38 600	44.3	28.5	149	13
G.	2	77 100	39 100	42.7	29	149	15
H.	1 7/16	77 300	39 300	41.0	29	153	15
I.	⅞	79 300	40 200	45.3	29.5	153	14
J.	7/16	79 500	40 500	47.2	30	156	14.5

[a] Lower yield point by drop-of-beam.

[b] Charpy V-notch at room-temperature (74 F).

PART IV.—SECTION SIZE EFFECT

A rating of the relative merits of cast and wrought steels in fatigue also requires that some consideration be given to the possible section size effect, since it is now recognized that the size of a component may influence its performance characteristics.

Whenever a large section shows poorer performance than a smaller prototype, the loss in properties is attributed to the so-called "section size effect." This effect has at least two components. The first is the geometrical phenomenon resulting from the mechanics of testing and is dependent upon increases in strain

TABLE VII.—TENSILE PROPERTIES AT VARIOUS LOCATIONS IN THE LONGITUDINAL AND TRANSVERSE DIRECTIONS OF A 6¼-IN. SQUARE SECTION OF WROUGHT C1030 STEEL (NO. 28) NORMALIZED FROM 1600 F AND TEMPERED AT 1200 F.

Location Code Letter	Distance from Surface, in.	Tensile Strength, psi	Yield Point,[a] psi	Reduction of Area, per cent	Elongation in 2 in., per cent	Brinell Hardness Number
			LONGITUDINAL			
A. . . .	½	74 100	43 700	64.3	35.5	146
B. . . .	1½	75 200	42 600	62.3	34	149
C. . . .	2½	74 600	42 300	56.3	33	143
			TRANSVERSE			
A. . . .	½	77 800	45 700	35.7	26.5	146
B. . . .	1½	79 800	45 200	36.2	24.5	149
C. . . .	2½	74 900	43 400	24.2	16	143

[a] Lower yield point by drop-of-beam.

TABLE VIII.—SECTION SIZE EFFECT DATA ON THE TENSILE, HARDNESS, AND IMPACT PROPERTIES OF CAST 8635 STEEL (NO. 29) NORMALIZED FROM 1600 F AND TEMPERED AT 1200 F.

Position Code Letter	Distance from Surface, in.	Tensile Strength, psi	Yield Stress, 0.2 per cent offset, psi	Reduction of Area, per cent	Elongation in 2 in., per cent	Brinell Hardness Number	Impact Energy,[a] ft-lb
			1¼-IN. SECTION SIZE				
A.	5/8	113 800	86 700	36.1	17	248	17
B.	5/16	115 700	89 400	35.9	17	248	16.5
			3-IN. SECTION SIZE				
C.	1 1/16	115 600	86 700	25.9	14.5	248	12
D.	3/4	117 700	86 700	29.7	15	255	13
E.	3/8	119 200	91 300	32.4	15.5	262	12.5
			6-IN. SECTION SIZE				
F.	2½	105 900	65 400	18.5	13.5	228	13.5
G.	2	108 200	66 800	21.5	14	235	13
H.	1 7/16	110 100	70 400	21.6	14.5	235	14.5
I.	7/8	114 300	75 400	21.9	14.5	248	13
J.	7/16	115 000	81 000	24.4	15.5	248	12

[a] Charpy V-notch at room-temperature (74 F).

TABLE IX.—SECTION SIZE EFFECT DATA ON THE TENSILE, HARDNESS, AND IMPACT PROPERTIES OF CAST 8635 STEEL (NO. 30) NORMALIZED FROM 1600 F, WATER QUENCHED FROM 1500 F, AND TEMPERED AT 1100 F.

Position Code Letter	Distance from Surface, in.	Tensile Strength, psi	Yield Stress, 0.2 per cent offset, psi	Reduction of Area, per cent	Elongation in 2 in., per cent	Brinell Hardness Number	Impact Energy,[a] ft-lb
			1¼-IN. SECTION SIZE				
A.	5/8	143 800	126 500	30.7	14.5	302	31
B.	5/16	147 400	131 900	33.7	15	311	29.5
			3-IN. SECTION SIZE				
C.	1/16	141 500	121 500	28.6	14	293	30.5
D.	3/4	145 000	127 500	29.2	14.5	302	28
E.	3/8	147 200	132 400	35.9	15	311	29
			6-IN. SECTION SIZE				
F.	2½	115 900	85 900	15.8	10.5	248	10.5
G.	2	117 400	87 200	17.4	11.5	248	10.5
H.	1 7/16	121 400	92 100	17.6	12	262	11
I.	7/8	126 600	100 300	24.0	13	269	10
J.	7/16	137 100	119 000	27.8	13.5	293	18 to 33

[a] Charpy V-notch at room-temperature (74 F).

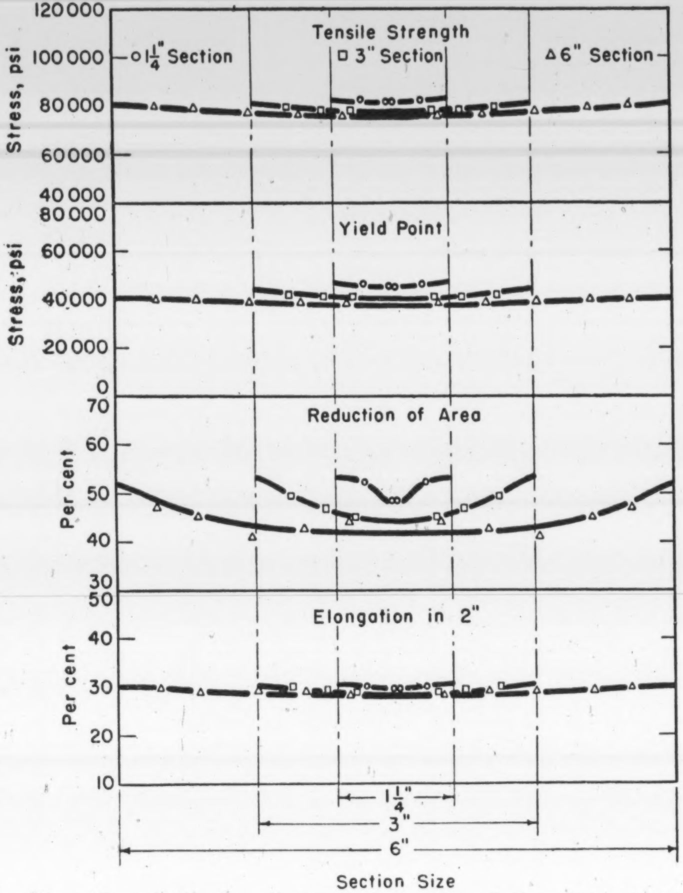

FIG. 14.—Distribution of Tensile Properties of Cast 1030 Steel. Normalized from 1600 F and tempered at 1200 F.

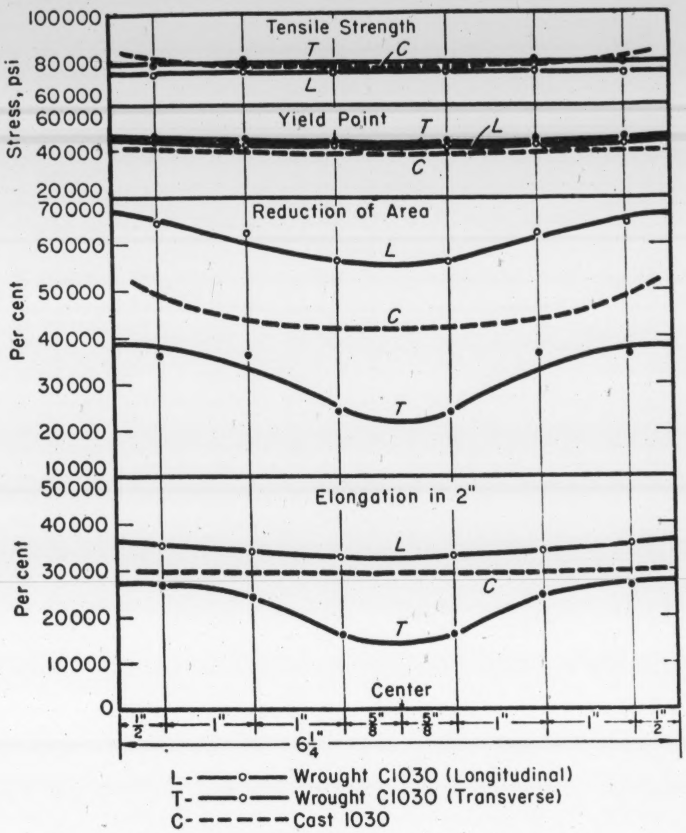

FIG. 15.—Distribution of Longitudinal and Transverse Tensile Properties of a $6\frac{1}{4}$ in. Section of Wrought C1030 Steel. Normalized from 1600 F and tempered at 1200 F. (Dotted curves are for a 6 in. section of cast 1030 steel with the same heat treatment.)

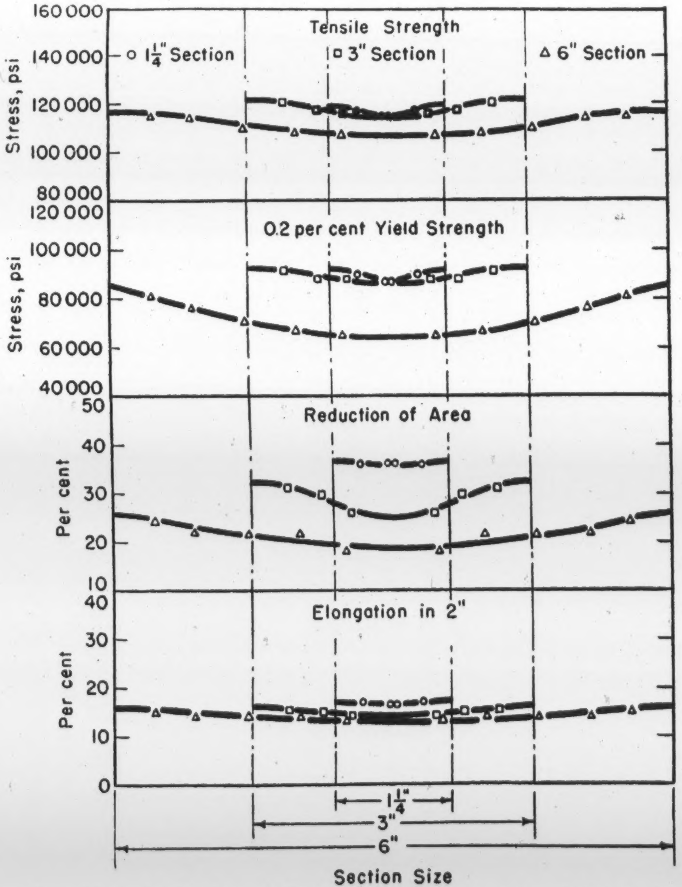

FIG. 16.—Distribution of Tensile Properties of Cast 8635 Steel. Normalized from 1600 F and tempered at 1200 F.

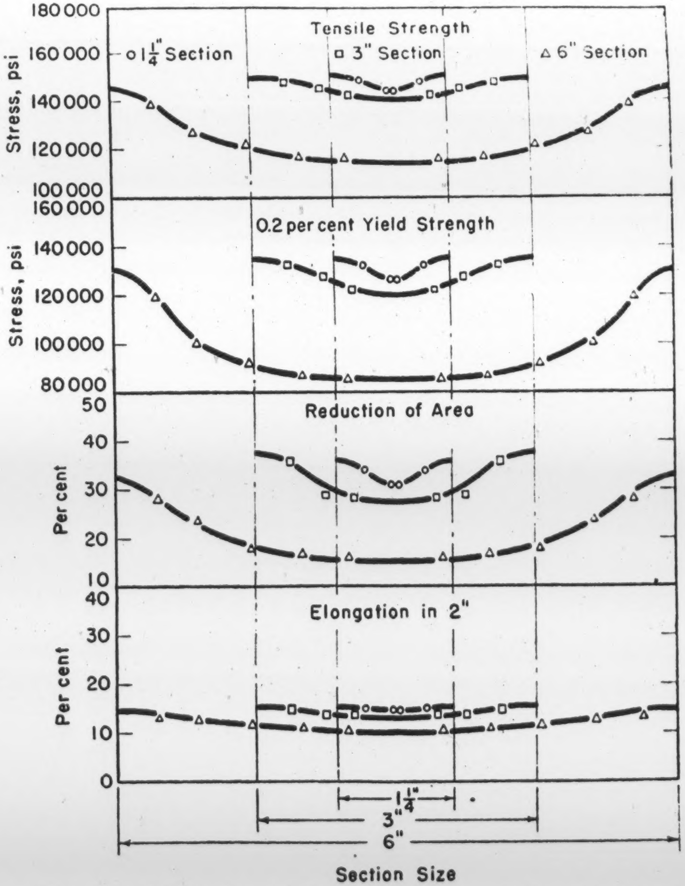

FIG. 17.—Distribution of Tensile Properties of Cast 8635 Steel. Water quenched from 1500 F and tempered at 1100 F.

gradients brought about by increases in the dimensions of the test specimens. The second—the mass or metallurgical phenomenon—arises from imperfections in the material and is dependent upon factors which present opportunities for possible stress concentration below the surface, such as inclusion content, compositional segregation, soundness, etc.

While both of the above phenomena would act together in producing a given size effect, a critical evaluation of the resultant behavior requires a separation of the two components.

An evaluation of the integrated size effect was not attempted because of equipment limitations; however, the mass component of the size effect for cast and wrought steels was studied by determining the distribution of properties throughout various section sizes using R. R. Moore test specimens of constant size.

Two different cast steels were investigated: a 1030 normalized-and-tempered and an 8635 normalized-and-tempered and quenched-and-tempered. The cast steels were heat treated at the foundry and supplied in three different section sizes, namely, 1¼-in., 3-in., and 6-in.

The study of wrought steel was limited to a 6¼-in. square hot-rolled bloom (2-ft-long section) of C1030 steel which was normalized and tempered in the laboratory. Specimens were taken from the longitudinal and transverse directions of the wrought steel to determine the effect of directionality. The method of obtaining specimens from various locations of the original sections is shown in Figs. 12 and 13 for cast steel and wrought steel, respectively.

In addition to the experimental work, a literature survey was made to determine the extent of section size effect in fatigue.

Tensile Properties:

The tension test results, Tables VI to IX, have been plotted for the various locations of each steel as shown in Figs. 14 to 17 to obtain the distribution of properties for each section size.

The tensile strength, yield point, and elongation curves in Fig. 14 for the normalized-and-tempered cast 1030 steel show a practically flat distribution for all sizes and slightly lower properties with increasing section size. However, the reduction of area distribution points out that, for each section, this property decreases as the center of the section is approached. Also, the over-all level of the distribution is lowered as the section size is increased. The per cent reduction of area, then, is more sensitive in depicting ductility variations than is the elongation criterion.

The distribution of the tensile properties for the 6¼-in. section of wrought 1030 steel is plotted for the longitudinal and transverse directions in Fig. 15 along with results obtained for the 6-in. section of cast 1030 steel. The tensile and yield strength distribution curves for the wrought steel (in both the longitudinal and transverse directions) and the cast steel were all relatively flat and essentially the same. The tensile ductility (reduction of area and elongation) curves indicate that the cast steel properties are intermediate to the tensile ductility value of the wrought steel in the longitudinal and transverse directions.

In Fig. 16 the distribution of tensile properties for the normalized-and-tempered cast 8635 steel reveal practically flat curves for the 1¼-in. size. The over-all distribution level of each property decreases with increasing size and each property decreases from the surface to the center of a section. Again, the reduction of area values more clearly show

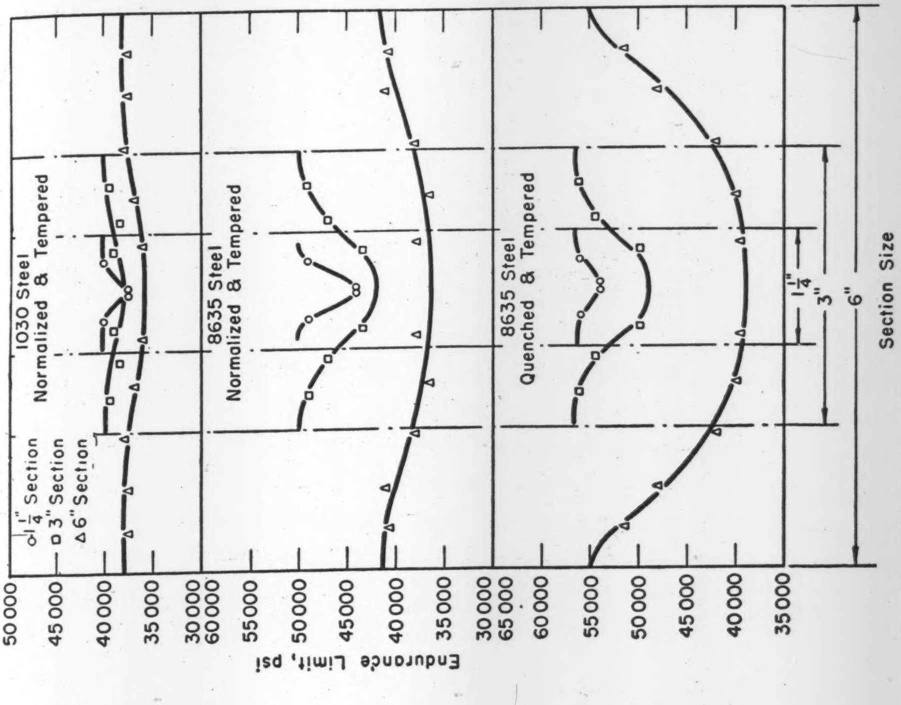

FIG. 19.—Distribution of Endurance Limit for Various Section Sizes of Cast Steels.

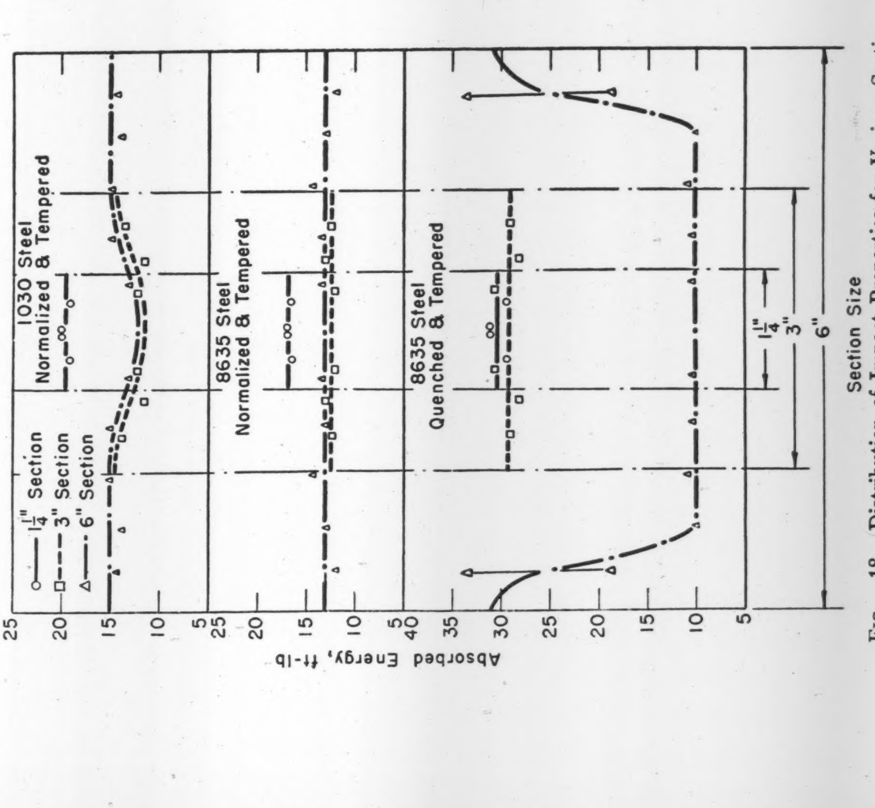

FIG. 18.—Distribution of Impact Properties for Various Section Sizes of Cast Steels. Charpy V-notch at 74 F.

ductility variations with section size than do the elongation values.

The curves in Fig. 17 for the quenched-and-tempered cast 8635 steel show the characteristic U-shaped distributions as the section size increases (for all tensile properties, except per cent elongation). The $1\frac{1}{4}$-in. and 3-in. sizes were essentially through-hardened, as evidenced by the high yield-tensile ratio. The 6-in. size hardened to a depth of about $\frac{5}{8}$-in. as noted from the fracture appearance of the test specimens at a position $\frac{7}{16}$ in. from the edge. The individual test results at this position showed a difference of about 9000 psi in tensile strength, which, along with a significant difference in the yield-tensile ratio, indicated that the depth of the essentially hardened zone was located close to a position $\frac{7}{16}$ in. from the edge. Again, the reduction of area is much more sensitive in revealing changes in ductility than is the elongation.

TABLE X.—SECTION SIZE EFFECT DATA ON THE FATIGUE PROPERTIES OF CAST STEELS.

Position Code Letter	Distance from Surface, in.	1030 Steel (No. 27) Normalized and Tempered		8635 Steel (No. 29) Normalized and Tempered		8635 Steel (No. 30) Quenched and Tempered	
		Endurance Limit, psi	Endurance Ratio	Endurance Limit, psi	Endurance Ratio	Endurance Limit, psi	Endurance Ratio
		$1\frac{1}{4}$-IN. SECTION SIZE					
A............	$\frac{5}{8}$	37 500	0.46	44 000	0.39	54 000	0.37
B............	$\frac{5}{16}$	40 000	0.49	49 000	0.42	56 000	0.38
		3-IN. SECTION SIZE					
C............	$1\frac{1}{16}$	39 000	0.50	43 500	0.38	50 000	0.35
D............	$\frac{3}{4}$	38 500	0.49	47 000	0.40	54 500	0.38
E............	$\frac{3}{8}$	39 400	0.50	49 000	0.41	56 000	0.38
		6-IN. SECTION SIZE					
F............	$2\frac{1}{2}$	36 000	0.47	38 000	0.36	39 500	0.34
G............	2	37 000	0.48	36 500	0.34	40 000	0.34
H............	$1\frac{7}{16}$	38 000	0.49	38 000	0.35	42 000	0.35
I............	$\frac{7}{8}$	37 500	0.47	41 000	0.36	48 000	0.38
J............	$\frac{7}{16}$	37 500	0.47	40 500	0.35	51 500	0.38

Although the hardness distributions have not been plotted, the results show that this property will parallel the tensile strength.

Impact Properties:

The room temperature impact properties for the various cast steels have been plotted for the various locations as shown in Fig. 18 to obtain the distribution for each section size. The data for these plots are found in Tables VII, VIII, and IX. The distribution curves for both the normalized-and-tempered 1030 and 8635 steels reveal that the $1\frac{1}{4}$-in. section has properties somewhat superior to those of the 3-in. and 6-in section sizes. No difference is apparent between the 3- and 6-in. sections. It should also be noted that for any given section size of either of these two steels the energy absorbed is relatively independent of the position throughout the section. In addition, the fracture appearance of the specimens from all positions of these two steels indicated that the temperature of testing was within the transition temperature range. Both steels evidenced a 20 to 30 per cent fibrous fracture throughout the $1\frac{1}{4}$-in. section and a 10 to 15 per cent fibrous fracture throughout the 3-in. and 6-in. sections.

The high impact properties obtained throughout the $1\frac{1}{4}$-in. and 3-in. sections for the quenched-and-tempered 8635 cast steel are characteristic of a martensitic structure. The fracture appearance of the specimens from all positions of these two sizes showed that the specimens were tested at a temperature somewhere along the upper branch of the transition curve. Low impact properties were obtained at all positions with the 6-in. section throughout the section with about a 5 to 10 per cent fibrous fracture, except at the position $\frac{7}{16}$ in. from the surface. The two tests at this location showed a wide variation in properties.

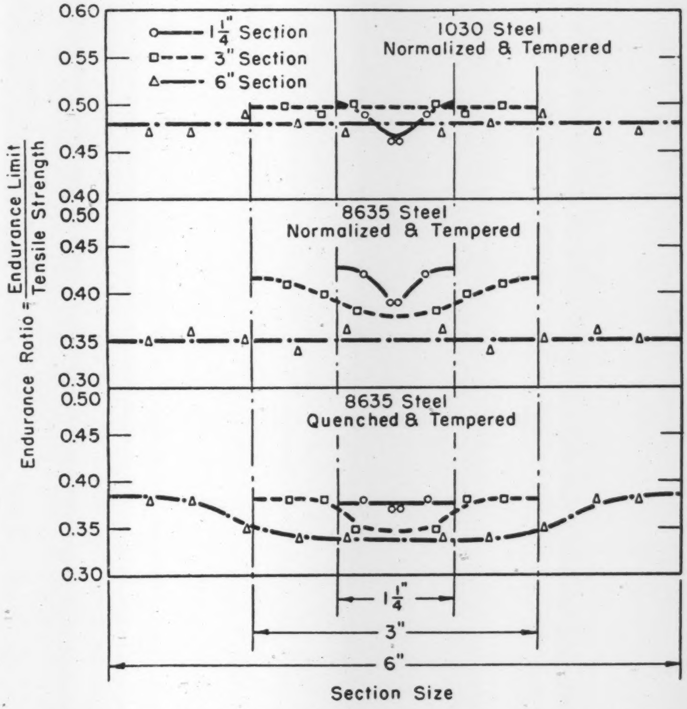

FIG. 20.—Distribution of Endurance Ratio for Various Section Sizes of Cast Steels.

The relatively low value of energy absorbed (18 ft-lb) was associated with about a 30 per cent fibrous fracture, while the high value (33 ft-lb) evidenced a more ductile fracture. This difference indicates that appreciable amounts of high-temperature transformation products are present near position J, that is, the depth of the essentially martensitic structure extends to about this point as previously noted with the tension tests.

Unnotched Fatigue Properties:

The fatigue results for the cast steels in Table X show, for a given steel and section size, a trend to lower properties in passing from the surface to the center of the section. The variation with steel and section size can more readily be seen in the distributions of endurance limit, shown in Fig. 19. These distribu-

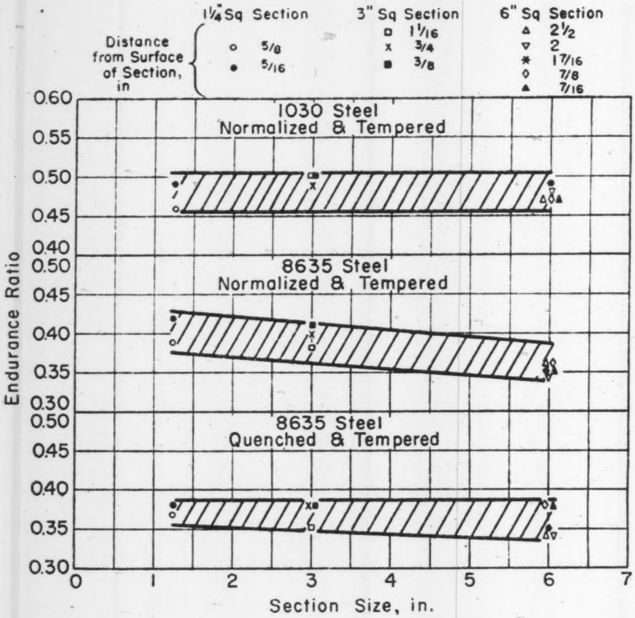

FIG. 21.—Range of Endurance Ratios of Small Specimens Obtained from Various Section Sizes of Cast Steels.

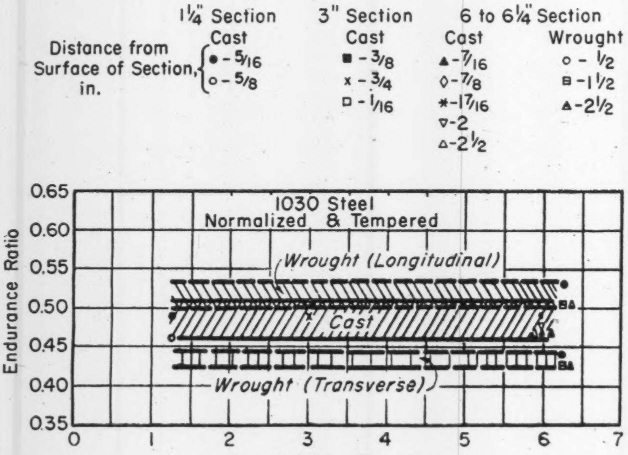

FIG. 22.—Range of Endurance Ratios of Small Specimens Obtained from Various Section Sizes of Cast and Wrought Steel.

TABLE XI.—FATIGUE PROPERTIES AT VARIOUS LOCATIONS IN THE LONGITUDINAL AND TRANSVERSE DIRECTIONS OF A 6¼-IN. SQUARE SECTION OF WROUGHT C1030 STEEL (NO. 28) NORMALIZED FROM 1600 F AND TEMPERED AT 1200 F.

Location Code Letter	Distance from Surface, in.	Tensile Strength, psi	Endurance Limit, psi	Endurance Ratio
LONGITUDINAL				
K	2½	74 600	38 000	0.51
L	1½	75 200	38 500	0.51
M	½	74 100	39 500	0.53
TRANSVERSE				
K	2½	75 300	32 500	0.43
L	1½	79 800	34 000	0.43
M	½	77 800	34 000	0.44

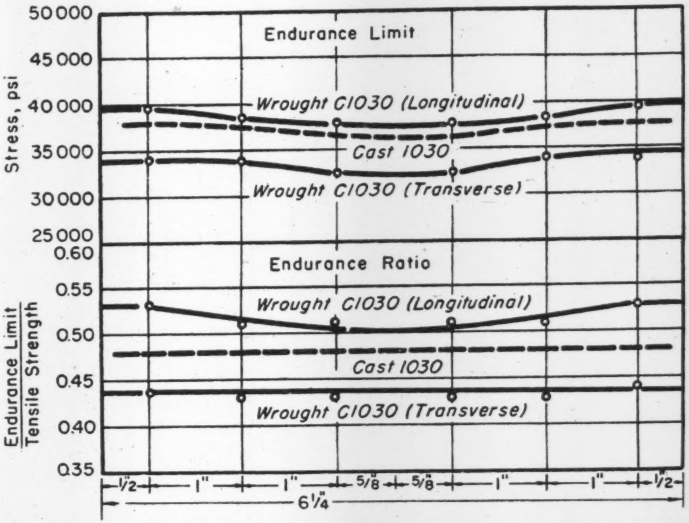

FIG. 23.—Distribution of Endurance Limit and Endurance Ratio in the Longitudinal and Transverse Directions of a 6¼ in. Section of Wrought C1030 Steel. Normalized from 1600 F and tempered at 1200 F. (Dotted curves are for a 6-in. section of cast 1030 steel with the same heat treatment.)

tions show that for a given steel the endurance limit varies in the same manner as the tensile strength.

If the endurance limit were dependent solely upon tensile strength, the endurance ratio (endurance limit divided by the tensile strength) would be a constant for a given steel and independent of the position in a section. However, a consideration of Table X and the distribution of ratios in Fig. 20 reveal that the ratio is somewhat dependent upon the position in a particular section. In general, the ratio tends to decrease slightly in passing from the surface to the center of section. Replotting the endurance ratio data, Fig. 21, shows graphically the range of values to be expected as a function of the section size. The indications are that for a given steel the lowest and highest values for a given section encompass a range which amounts to about a 10 per cent difference in endurance ratio. It should be noted that for the normalized-and-tempered 8635 steel both the maximum and minimum values appear to decrease slightly with increasing section size. This is not readily apparent with either the normalized-and-tempered 1030 steel or the quenched-and-tempered 8635 steel. As a generalization, the minimum and the maximum endurance ratios are to be found at the center of the 6-in. section and the surface of the $1\frac{1}{4}$-in. section, respectively. This range of minimum to maximum ratios is 0.46 to 0.50 for the 1030 steel; 0.34 to 0.42 for the normalized and tempered 8635 steel; and 0.34 to 0.38 for the quenched-and-tempered 8635 steel.

Among the factors that may have contributed to the slight decrease in endurance ratio with increasing section size, the following may be mentioned: (1) increase in grain size, (2) increase in size of inclusions, and (3) microstructural changes and microstructural inhomogeneity.[5] It is difficult to isolate any one as the major contributor because of the small changes in endurance ratio involved.

The slight reduction in endurance ratio found in passing from the surface to the center of a given section would probably be of negligible influence on the performance of the whole section in bending fatigue. The outer fibers generally receive the maximum stress in service, and in view of the fact that the applied stress below the surface drops off rapidly it would appear that the properties at the edge of the section largely dictate the over-all performance.[6] A consideration of the properties near the surface (top boundary line of the range shown in Fig. 21) indicates that only the endurance ratio of the normalized-and-tempered 8635 steel is somewhat dependent on the section size.

The endurance ratio data for the wrought steel have also been replotted, Fig. 22, to show graphically the range of values to be expected as a function of the section size. The results with the cast 1030 steel are also shown. Although data are available only for the distribution of endurance ratio throughout a $6\frac{1}{4}$-in. section of wrought 1030 steel, the same range of values for the longitudinal direction (0.51 to 0.53) and the transverse direction (0.43 to 0.44) has been extended to include smaller section sizes. This extrapolation seems reasonable because the range of values for this heat and size is as high as any wrought steel of this particular grade reported in the literature for smaller sections, at least in the longitudinal direction. Along these same lines, it should be noted that the range of values shown in Fig. 22 pertain to a single heat of steel. The results of preliminary investigation indicated that the endurance ratio for commercial grades of wrought steel in the 0.30 to 0.40 per cent carbon range obtained from small sections in the longitudinal direction can be as low as 0.44 and as high as 0.53.

The summary fatigue test results for the wrought steel are shown in Table XI and indicate that at every location under test the longitudinal properties are superior to those in the transverse direction. This superiority which amounts to about 20 per cent can be seen in the distributions of endurance limit and endurance ratio in Fig. 23. These data also show that for a given direction for the wrought steel the fatigue properties tend to be slightly lowered in passing from the surface to the center of the section.

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[5] A study with wrought 4140 steel (4) on the effects on non-martensitic products of transformation on the properties of tempered martensite revealed that a structure containing, (*a*) 5 per cent peralite and 8 per cent ferrite, or (*b*) 50 per cent bainite lowered the endurance ratio about 15 per cent. The latter structure would be approximated by position I in the quenched-and-tempered 8635 steel; however, the endurance ratio obtained at this position (0.38) was the same as that for a completely martensitic structure.

[6] A possible exception to this generalization would be the example of a section with high strength properties at the surface which drop off very rapidly immediately below the surface (for example, a shallowed hardened or case hardened section). Although the endurance ratio may be the same at the surface and at the junction of the core, the endurance limit at the surface would be much higher due to the higher tensile strength. Then, even though the applied stress gradient is steep in this region, the endurance limit gradient may be even steeper, and a region may exist (below the surface) where the applied stress would be greater than the endurance limit, resulting in sub-surface failure. Although not conclusive, this reasoning may apply to the improvement of the fatigue properties of large, surface-hardened wrought steel bars brought about by increasing the depth of the hardened case (**5**).

The distribution of fatigue properties previously obtained with a 6-in. section of cast 1030 steel is shown in Fig. 23 to be intermediate to the longitudinal and transverse distributions for the wrought C1030 steel. This comparison suggests that the fatigue properties must be somewhat dependent upon the ductility because, although the strength properties are essentially the same (Fig. 15), a pronounced difference is evident in the reduction of area values. In fact, the fatigue properties are rated in the same order as the tensile ductility.

TABLE XII.—LONGITUDINAL AND TRANSVERSE FATIGUE PROPERTIES OF TWO LOW-ALLOY STEEL FORGINGS (VON ROSSING).

Materia	Composition, per cent				Dimensions, in.	Longitudinal		Transverse	
	Carbon	Chromium	Molybdenum	Nickel		Tensile Strength, psi	Endurance Ratio	Tensile Strength, psi	Endurance Ratio
Cr-Mo steel..........	0.37	1.19	0.26	0.14	4 by 7	Surface...... 112 100	0.59	112 100	0.54
						Center...... 109 600	0.53	109 600	0.53
Ni-Cr-Mo steel......	0.34	0.57	0.45	1.60	15 by 15	Surface...... 110 300	0.47	109 900	0.33
						Center...... 107 800	0.39	108 400	0.32

Other Investigations:

As previously pointed out, the section size effect in fatigue consists of at least two components—the mass effect and the geometric effect. The present investigation has contributed experimental data on the former, and the results may be compared with those reported previously by other investigators. While the geometric component has received no attention *per se*, a limited amount of fatigue data is available in the literature on the integrated section size effect which is also reviewed. The data in the literature refer only to wrought steel.

Mass Component:

Von Rossing (6) determined the longitudinal and transverse fatigue properties of unnotched specimens from the surface and the center of two low-alloy steel forgings. The results are summarized in Table XII. These data show that the longitudinal fatigue properties are 10 to 15 per cent lower for specimens from the center than those machined from material near the surface. For the transverse direction, the surface and center properties are about the same. In addition, the directionality effect for the small forging was about 10 per cent, whereas this effect for the larger forging was of the order of 25 per cent.

Of special interest is the fact that the level of the endurance ratio of the smaller forging was as high as obtained in the present investigation with specimens from small wrought sections.

Integrated Section Size Effect.—Many investigators have studied the integrated section size effect for the longitudinal direction of wrought steel, that is, the effect of test specimen diameter of the fatigue properties, without separating the mass and geometric components of this effect.

Moore and Morkovin (8) and Moore

(9) indicated that the size effect for unnotched 1020, 1035, 2345, and X4130 steels disappears at a relatively small absolute dimension; the endurance limit decreases about 10 to 15 per cent as the section increases from $\frac{1}{8}$ in. to $\frac{1}{2}$ in., then remains relatively constant for larger sections up to 2 in. These same steels with a notch also showed a size effect. Dolan and Handley (10) and Horger and Maulbetsch (11) also found that the unnotched fatigue properties for small specimens were 10 to 15 per cent higher than for large specimens when testing in the size range from $\frac{1}{8}$ to $1\frac{3}{4}$ in.

Two other investigations would indicate that the size effect is still greater with larger specimens. Buchman (12) investigated a mild steel and found about a 40 per cent reduction in properties when the diameter was increased from 0.60 in. to 4 in. Horger and Neifert (13) found that 6-in. diameter specimens had a minimum endurance limit 35 per cent lower than that for 0.3-in. diameter specimens from as-forged and also from normalized stock of plain-carbon steels in the 0.40 to 0.50 per cent carbon range. However, as pointed out, "This 35 per cent reduction cannot be attributed to section size *per se*. Several additional factors such as structural variations, carbon content, microstructure, and surface conditions also influence this reduction in endurance limit." It should also be mentioned that the authors were testing steels whose tensile strength varied as much as 20,000 psi. This variance in tensile strength could of course be reflected in the fatigue properties. In addition, different types of specimens were used (R. R. Moore and cantilever beam) whose dimensions were not strictly proportional.

This review serves to emphasize that the available information on the integrated section size effect is meager and inconclusive. It also points out that the commonly applied allowance of 20 per cent for the section size effect in fatigue for wrought steel may be in serious error for large sections.

SUMMARY

The relative fatigue properties of comparable cast and wrought steels in the 0.30 to 0.40 per cent carbon range were determined, employing R. R. Moore test specimens. The work was presented in four parts:

Part I—Effect of Steel Composition and Heat Treatment:

A plain-carbon series steel was tested in the annealed and in the normalized-and-tempered condition; four low-alloy series steels were tested in the normalized-and-tempered and in the quenched-and-tempered condition.

The unnotched endurance limit of any one of the cast steels was about $\frac{4}{5}$ the endurance limit of the corresponding wrought steel. For the plain-carbon series steel the endurance ratio was 0.40 for the cast steel and 0.48 for the wrought steel. For the four low-alloy series steels the endurance ratio was found to be 0.42 to 0.50 for the cast steels as compared to the 0.55 to 0.60 range for the wrought steels.

With the introduction of a notch, the endurance limit for both cast and wrought material was significantly affected, with the cast steel exhibiting a greater notch resistance. The notched endurance limit for the cast and the wrought steel for any given series and heat treatment was about the same, being 0.27 to 0.32 times their respective tensile strengths.

Part II—Effect of Surface Finish:

Cast 8640 and wrought 8640 steels were tested in the normalized-and-tempered condition. For a polished finish, the endurance ratio for the cast steel (0.45) was about $\frac{4}{5}$ that of the wrought steel (0.59); with a lathe-turned finish the endurance ratio for the cast steel was unaffected whereas the ratio for the wrought steel suffered a pronounced decrease to 0.46. Cast-to-shape fatigue specimens tested with an as-cast finish effected a considerable decrease in the fatigue properties, the endurance ratio being lowered to 0.32. This ratio was the same for specimens made with and without risers, thus indicating no effect of centerline shrinkage.

Part III—Directionality:

With unnotched specimens, the deleterious effect of directionality for a 4-in. round of wrought 4140 steel was shown by a 13 per cent decrease in the endurance ratio from 0.55 for the longitudinal direction to 0.48 for the transverse direction. Data in the literature indicate that this reduction can be as high as 30 per cent for dirty steels. With the introduction of a notch, the endurance limit in both directions was significantly affected. The notched endurance ratio was 0.25 for the longitudinal direction as compared with 0.24 for the transverse direction.

Part IV—Section Size Effect:

Small unnotched specimens of constant size were taken from various locations in 1¼-in., 3-in., and 6-in. square sections of each of the following cast steels: (*a*) normalized-and-tempered 1030, (*b*) normalized-and-tempered 8635, and (*c*) quenched-and-tempered 8635. The fatigue data were supplemented by regular tensile and Charpy V-notch impact tests at room-temperature.

The distribution curves revealed that for a given steel the endurance limit varied in the same manner as the tensile strength. However, the endurance limit was not dependent solely on the tensile strength. For each of the three steels, the endurance ratio was slightly lowered in passing from the surface to the center of a given section. In addition, the endurance ratio values for the normalized-and-tempered 8635 steel decreased slightly as the section size increased; the other two steels were relatively insensitive to increasing section size. In general, the lowest ratio was obtained at the center of the 6-in. section and the highest ratio at the surface of the 1¼-in. section. This range of minimum to maximum endurance ratio was 0.46 to 0.50 for the 1030 steel; 0.34 to 0.42 for the normalized-and-tempered 8635 steel; and 0.34 to 0.38 for the quenched-and-tempered 8635 steel.

A limited study of the section size effect for wrought steel was carried out with a 6¼-in. square hot-rolled bloom of C1030 steel. Little change in the fatigue properties was experienced in passing from the surface to the center of the section for a given direction; however, anisotropy was marked. The range of endurance ratios was 0.51 to 0.53 for the longitudinal direction and 0.43 to 0.44 for the transverse direction. The distribution of the endurance ratio of the comparable cast 1030 steel was intermediate to the longitudinal and transverse distributions.

A review of the literature on the section size effect in fatigue for wrought steel suggested the following:

1. The mass component of this effect had an appreciable damaging influence only in the range of very large sections.
2. The combined mass and geometric components of the size effect may be as high as 40 per cent for large sections.

Acknowledgments:

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DISCUSSION

Mr. Evan H. Schuette.[1]—We are often misled by an undue emphasis on "notch sensitivity" when a more proper comparison might be the degree by which the "un-notched" strength exceeds the "notched" strength. If only "notch sensitivities" are compared in the present paper, the conclusion is that the wrought metal is inferior to the cast. Actually, a guess could be hazarded that the two materials are equally sensitive to notches (they show equal strength when notched), but that the cast material is influenced by some kind of stress raiser in its virgin state, so that its "un-notched" strength does not reach that of the wrought metal.

A similar phenomenon is usually observed when comparing longitudinal and transverse fatigue strengths of extruded metals. Plain bars will show the longitudinal direction to give the higher fatigue strength. When notched, however, the two directions are indistinguishable. It is hypothesized that such internal stress raisers as are present are greatly attenuated in the process of extrusion, so that their effect in the longitudinal direction is virtually eliminated. In the transverse direction, however, they continue to act as notches. There is presumably no difference in the real sensitivity to notches in the two directions.

Mr. L. J. Ebert (*author*).—The subject under discussion is one which is open to controversy, certainly. The interpretation put upon the data is perhaps valid. This has been discussed in some detail in our own laboratory but we have not given the matter sufficient consideration to state a positive position.

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[1] Dow Chemical Co., Midland, Mich.

THE EFFECT OF CHANGING CYCLIC MODULUS ON BENDING FATIGUE STRENGTH*

By A. A. Blatherwick[1] and B. J. Lazan[1]

The literature on fatigue contains many references to the nebulous size and shape factors. Laboratory data have demonstrated that both the shape and size of a specimen, particularly under nonuniform stress, may significantly affect its fatigue strength. The purpose of this paper is to study the possible relationships of stress redistribution during fatigue loading to the shape effects, and to examine the influence of type of test on fatigue strength.

An indication that the stress distribution may change during fatigue tests has been observed in recent years by several investigators. In rotating cantilever tests on mild steel (**1, 2**)[2] the ratio of cyclic stress to cyclic strain (the cyclic secant modulus) was observed to change under sustained fatigue stress. For some materials the cyclic modulus[3] decreases during the fatigue test (**2**), whereas for others, it was observed to increase (**3, 4**). Duce (**5**) observed this same behavior in a number of different materials under torsional fatigue stress. In fact he noted that under certain conditions some materials have a decreasing modulus followed by a later increase. Recent axial-stress tests at the University of Minnesota (**6**) on mild steel show similar results.

These observations imply that the stress distribution may change significantly under sustained cyclic stress in a manner which is characteristic of the material and the cross-section shape. The influence of cross-section shape on the relationship between static bending moment and strain has been demonstrated (**7**). However, the study of this effect under cyclic stress has not received its due attention.

This paper is concerned with both the analytical and experimental study of this problem. First hypothetical stress-strain relationships are assumed to simulate the behavior of real materials, and on the basis of these assumed properties, for which the cyclic secant modulus varies according to convenient laws, the fatigue behavior is calculated. Comparisons are then made between the fatigue strengths to be expected from various types of simulated bending fatigue tests. In each of these simulated test conditions, various shapes of cross-section are considered, and the influence of shape is discussed. This paper also includes a partial experimental check of some of the conclusions resulting from the analytical work.

Cyclic Stress-Strain Properties

In general, there appear to be two main types of material as far as response to cyclic stress is concerned. One type exhibits a gradually increasing stress amplitude with repeated cycles of constant strain amplitude while the other has a decreasing stress, at least in the early stages of fatigue life. The first type will be referred to in this paper as a "strain-hardening" material; the second type will be called "strain-softening."

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1033.

[3] The term "dynamic modulus of elasticity" used in previous work ((**1, 2, 3, 4**) will henceforth be referred to as the "cyclic secant modulus." It is defined, specifically, as the ratio of the maximum stress to the maximum strain (not necessarily at the same point) of the cyclic stress-strain hysteresis loop.

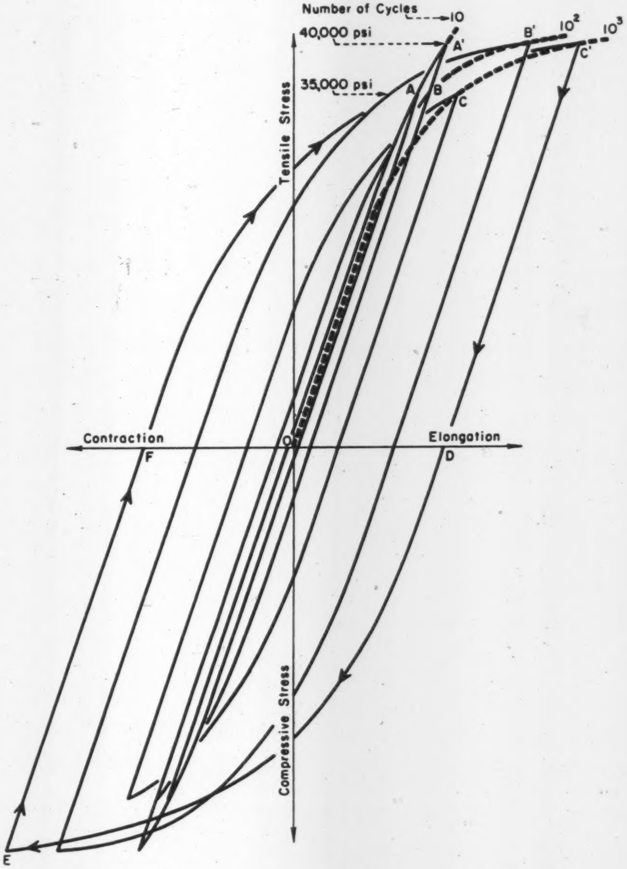


FIG. 1.—Stress-Strain Hysteresis Loops and Locus Curves for Mild Steel after Various Numbers of Cycles.

Most materials apparently belong to the strain-hardening type, but a few, of which mild steel is a notable example, belong to the second type which exhibit strain softening in the early stages of fatigue. For conciseness, this paper is concerned primarily with the latter type.

Figure 1 shows two sets of hysteresis loops at different stress amplitudes, for SAE 1020 steel under axial loading (6). Each set has one loop after 10 cycles, one after 100 cycles, and one after 1000 cycles of repeated stress. For the outer loop, which is typical, the arrows mark the direction of load application. Starting at the upper point *C′*, the curve is traced through zero stress at *D* to maximum compression at *E*, thence back to zero stress at *F*, and finally back to maximum tension at *C*′. Some portions of the curves are omitted in order to avoid confusion of too many intersections.

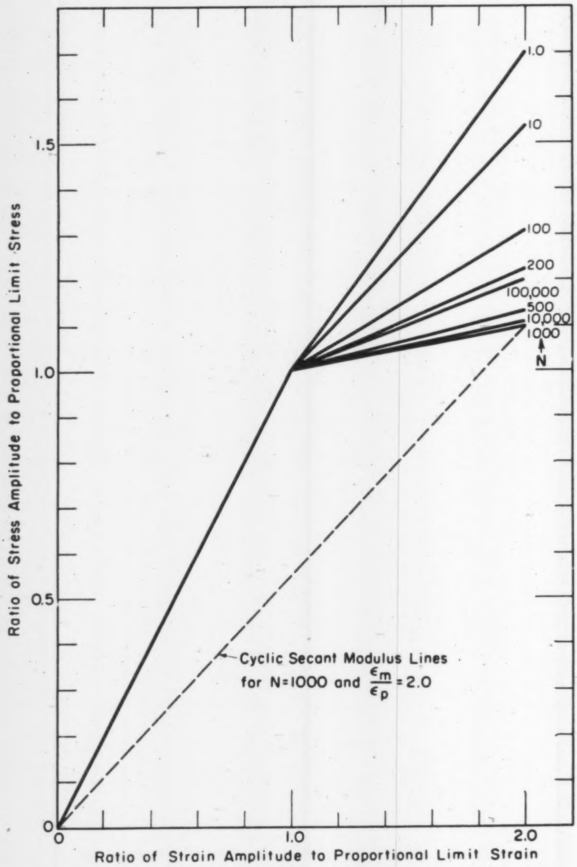


FIG. 2.—Family of Hypothetical Cyclic Stress-Strain Locus Curves for a Strain-Softening Material.

The softening effect is evident from the widening of the loops with increasing number of cycles. The dashed curves *OAA*′, *OBB*′, and *OCC*′, which are the loci of the maximum points of the hysteresis loops after 10, 100, and 1000 cycles respectively, will be referred to hereafter as the "cyclic stress-strain locus curves," or simply "locus curves," for the indicated number of cycles. In this work, these locus curves provide the relationship between the maximum values of cyclic stress and strain.

In order to study the effects of the changing cyclic modulus without the complication of additional factors necessarily arising in experimental work, hypothetical cyclic stress-strain relationships will now be employed to simulate the behavior of actual materials. For simplicity of calculations, cyclic stress-strain locus curves consisting of two straight-line segments are assumed.

The slope of the elastic leg remains constant under cyclic stress and the proportional limit remains fixed. The slope of the upper (plastic) leg, however, varies with N, the number of repeated cycles. For a strain-softening material, this slope decreases, while in the case of strain-hardening materials it increases.

This variation of slope may be expressed mathematically by multiplying the elastic modulus, E, by a function of N. A suitable function for a strain-hardening material is:

$$F_1(N) = 1 - e^{-\lambda(N+N_0)} \quad \ldots\ldots. (1)$$

in which λ and N_0 are parameters which give the desired initial conditions and regulate the rate of approach to the elastic condition. For a strain-softening material, the function used is:

$$F_2(N) = 1 - \gamma N^q e^{-\lambda N} \quad \ldots\ldots. (2)$$

where γ, q, and λ are suitable parameters chosen so as to approximate the behavior of mild steel which was studied experimentally.

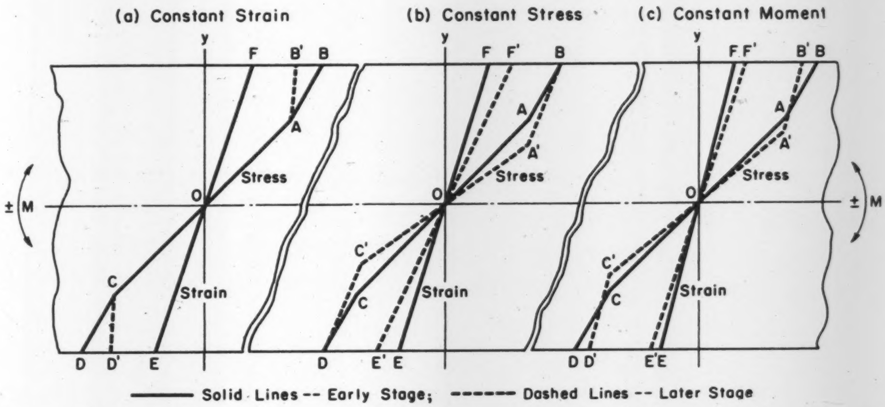


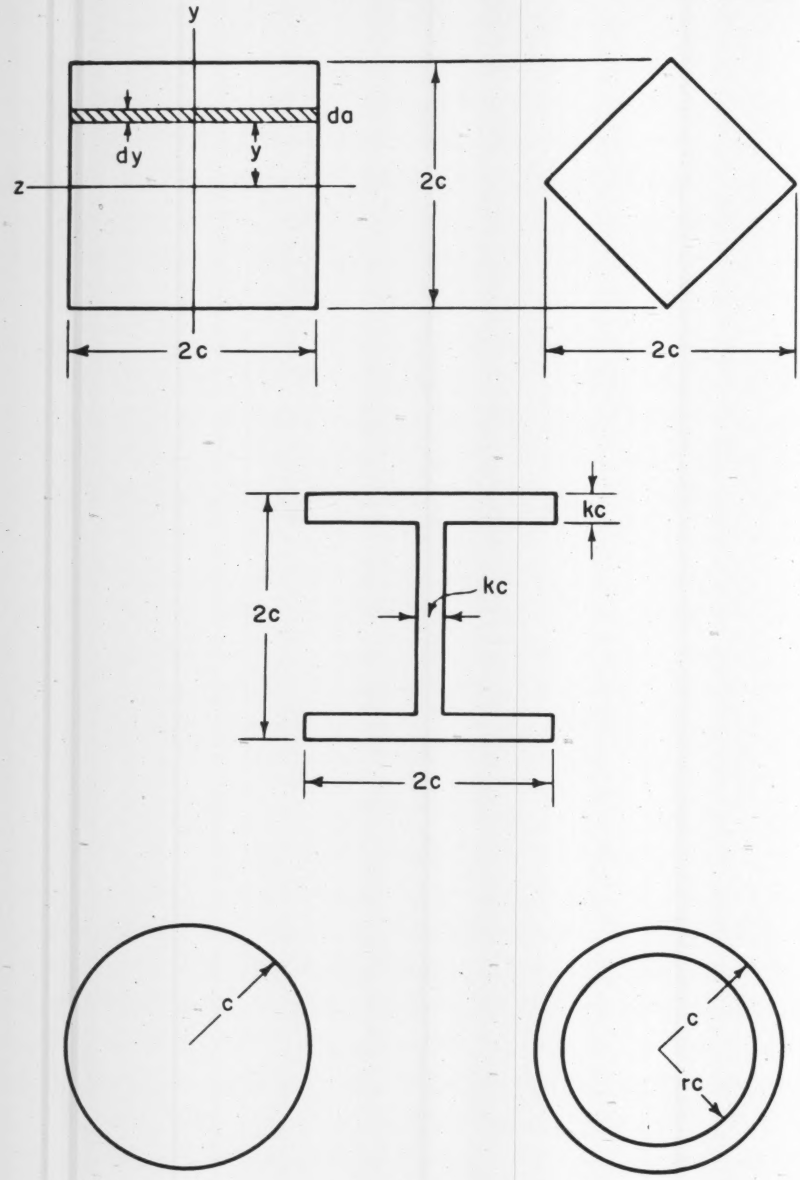
FIG. 3.—Stress and Strain Distributions in Specimen of a Strain-Softening Material for Three Different External Loading Conditions.

Figure 2 shows the set of locus curves for a strain-softening material obtained by use of function F_2 above. Only the tension half of the curves is shown, the compression portion being assumed identical. This assumption was verified experimentally for mild steel in axial stress tests. Also shown in the figure is the cyclic secant modulus line for $N = 1000$ and a strain of twice the proportional limit value. Each of the locus curves gives the stress-strain amplitude relationship for the pertinent number of cycles which is labelled at the upper end of the curve. It will be noted that the slope of the upper leg gradually decreases with increasing cycles up to 1000 after which it slowly rises. This is similar to the observed behavior of mild steel to be described later.

A similar set of locus curves for a strain-hardening material was constructed, but to conserve space it is not included in this paper. These curves, together with all others for strain-hardening materials are given in reference (8).

BENDING MOMENT, STRESS, AND STRAIN ANALYSIS

Aside from the material behavior discussed above and the usual assumptions generally employed in elementary beam



Note: r + k = 1

FIG. 4.—Cross-Sections of the Five Specimens Analyzed.

theory, three additional assumptions are made in this analysis:

1. Any plane cross-section before bending remains plane after bending occurs, even in the inelastic range.

2. No reaction occurs between adjacent filaments of the beam, and

3. A unique stress results from a given strain after a given number of cycles (either in axial loading or in bending).

The variable material properties discussed previously may significantly affect the non-linearity of bending stress and its distribution during fatigue tests. Figure 3 gives a qualitative indication of the changes that may be expected under different conditions for a strain-softening material. In Fig. 3(*a*) a longitudinal section of a portion of a specimen is shown. At a given stage of the test, the stress distribution is given by lines *DCOAB* and the strain distribution by *EOF*. If the test conditions are such that constant strain amplitude is maintained in the outer fibers, the stress distribution at a later stage will be *D'COAB'*. The strain distribution will, of course, remain the same. The decreased stress in the plastic ranges *AB'* and *CD'* will obviously cause a decrease in the bending moment.

A case of constant stress is represented in Fig. 3(*b*). Initially the stress distribution is indicated by lines *DCOAB* and the strain by *EOF*. After a number of cycles resulting in strain softening, the dashed lines *DC'OA'B* represent the stress values,[4] and in order to allow the stress to remain constant at *D* and *B*, the strain has increased to the line *E'OF'*. In this case, the bending moment has increased as a result of the higher stress throughout the specimen even though the maximum stress remains constant.

A constant-moment condition is represented in Fig. 3(*c*). The solid lines *DCOAB* show the distribution of stress and line *EOF* that of strain at one time in the test. Later, because of the cyclic strain softening, the stress distribution is as indicated by the dashed lined *D'C'OA'B'*. Since the bending moment is constant, the decreased stress in the outer portion must be accompanied by an increase of the stress in the inner region. In order that those fibers in the elastic range may exert more stress, the strain must be increased as shown by line *E'OF'*.

Moment Equations:

Figure 4 shows the cross-sections of the five specimen types to be studied. For the square section (upper left) the coordinate system is shown, along with a typical strip element, *da*. Now, for a given strain in the outer fibers, ϵ_m, the bending moment:

$$M = 2\int_0^c S(y)y\,\mathrm{d}a \qquad (3)$$

If the first moment of that part of the cross-sectional area whose height is *y* and whose base is along the *z* axis is designated by *m*, then since *m* is a function of *y*,

$$y\,\mathrm{d}a = \mathrm{d}m = \frac{\mathrm{d}m}{\mathrm{d}y}\,\mathrm{d}y \qquad (4)$$

Because of its relation to the shape of the section and its attendant role in determining response of a given cross-sectional shape to cyclic loading, $\frac{\mathrm{d}m}{\mathrm{d}y}$ will be referred to as the "shape function" and will be designated by:

$$\phi(y) = \frac{\mathrm{d}m}{\mathrm{d}y} \qquad (5)$$

Substituting from Eqs 4 and 5 into Eq 3 there results:

$$M = 2\int_0^c S(y)\phi(y)\,\mathrm{d}y \qquad (6)$$

The stress may be expressed in terms of *y*, using the assumed linear strain relationship:

$$\frac{\epsilon}{\epsilon_m} = \frac{y}{c} \qquad (7)$$

And writing the equations for stress

---

[4] Point *A* has moved parallel to the y-axis to position *A'*, and point *C* has likewise moved to *C'*, since the proportional limit of the assumed cyclic stress-strain curves remains at the same stress level.

from the assumed stress-strain locus curves:

$$S = E\epsilon, \quad \text{for} \quad 0 \leqq \epsilon \leqq \epsilon_p \ldots (8)$$

$$S = E\epsilon_p + EF(N)(\epsilon - \epsilon_p),$$

for $\epsilon_p \leqq \epsilon \leqq \epsilon_m$

where ϵ_p is the strain at the proportional limit. By substituting from Eqs 7 and 8 into Eq 6 and designating the ratio $\frac{\epsilon_p}{\epsilon_m}$ by p, the following expression for the bending moment is obtained:

$$M = 2E\ \frac{\epsilon_m}{c} \int_0^{pc} \phi(y) y\, \mathrm{d}y + 2E \int_{pc}^{c} \left[\epsilon_p + F(N)\left(\frac{\epsilon_m}{c}\, y - \epsilon_p\right)\right] \phi(y)\, \mathrm{d}y \ldots (9)$$

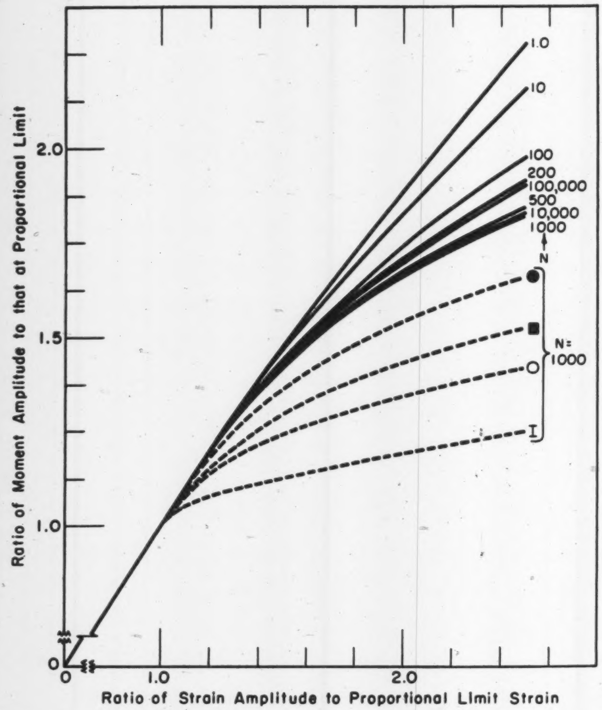


FIG. 5.—Moment Strain Curves for Diamond Cross Section, and 1000 Cycle Curves for the Four Other Shapes—Strain Softening Material.

As an example of the application of Eq 9, the case of the square cross-section will be illustrated. The developments for the other sections, omitted here to conserve space, are given in reference (8).

For the square section, $\phi = 2cy$ and Eq 9 becomes:

$$M = 4E\epsilon_m \int_0^{pc} y^2\, \mathrm{d}y + 4E\epsilon_m c \int_{pc}^{c} \left[\epsilon_p + F(N)\left(\frac{\epsilon_m}{c}\, y - \epsilon_p\right)\right] y\, \mathrm{d}y \,..(10)$$

The integral may now be evaluated, since $F(N)$ is constant with respect to the variable of integration. Upon integration, Eq 10 gives:

$$M = \frac{2}{3}\, Ec^3 \{2p^3\epsilon_m + 3[1 - F(N)]\epsilon_p (1 - p^2) + 2F(N)\epsilon_m(1 - p^3)\} \,..(11)$$

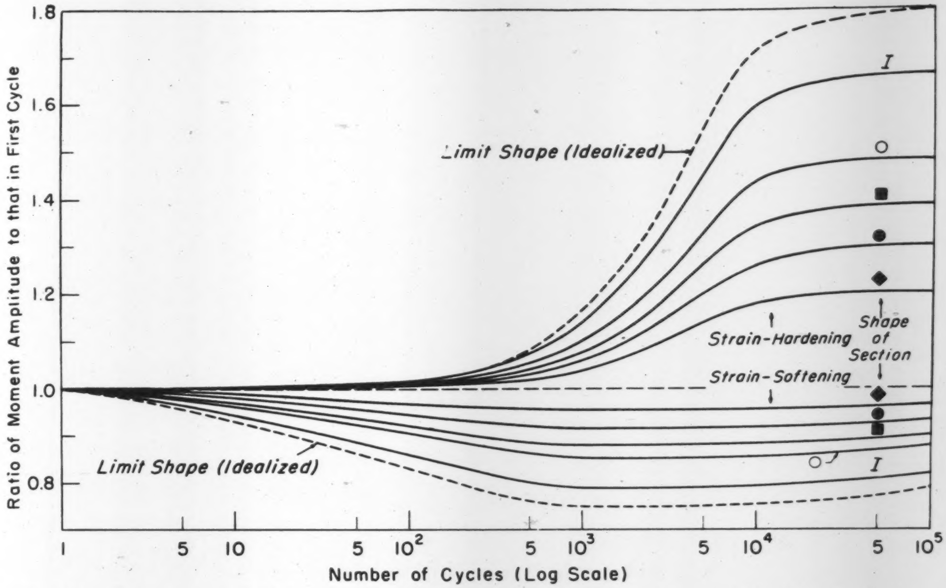


FIG. 6.—Moment *versus* N for the Five Specimen Shapes Under Constant-Strain Amplitude.

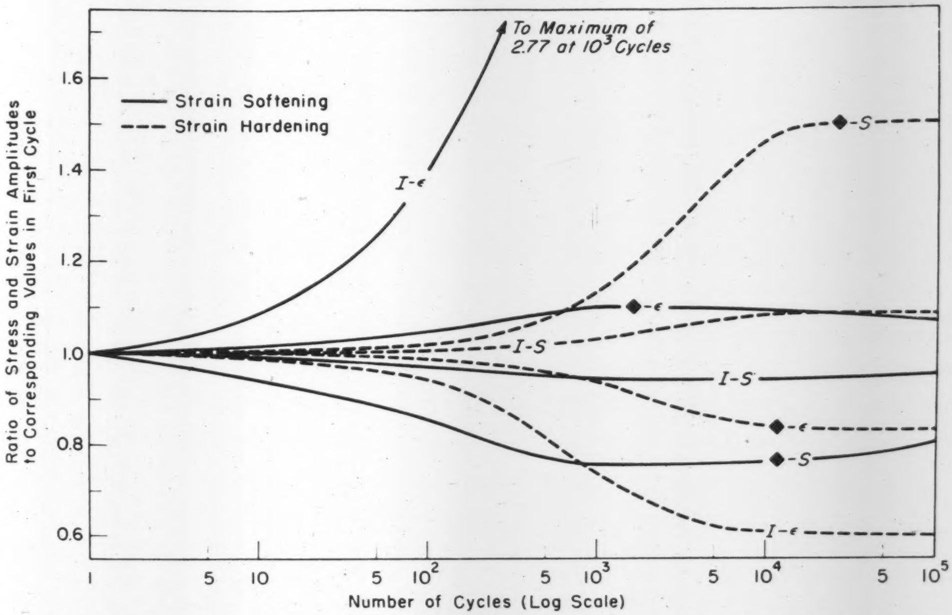


FIG. 7.—Stress and Strain Variation for Diamond and I Shapes of Both Strain-Hardening and Strain-Softening Materials Under Constant Moment Amplitude.

It will be noted that M depends upon the strain in the outer fibers, ϵ_m, and on the ratio of ϵ_p to ϵ_m, which together with E, is the material property.

Up to this point, no conditions have been imposed on the moment expression due to the cyclic modulus behavior; thus the above expression is valid for either a strain-hardening or a strain-softening material.

Moment-Strain Curves.—The moment equations for the individual cross-sectional shapes may now be calculated. For a strain-hardening material, substitution of Eq 1 in the moment expressions for each shape yields moment equations from which moment-strain curves may be plotted. The equations for a strain-softening material may be obtained by substituting Eq 2 in the same moment equations. Figure 5 shows a family of moment-strain curves for a diamond cross-section, for a strain-softening material. The dashed curves, for the other shapes at 1000 cycles, are included for comparison. In this figure the ratios of the moment amplitudes to that at the proportional limit are plotted as ordinates, while the ratios of strain amplitudes to the proportional-limit strain are the abscissae.

ANALYSIS OF BEHAVIOR UNDER VARIOUS FATIGUE CONDITIONS

From the cyclic stress-strain locus curves of Fig. 2 and similar ones for strain-hardening materials, and using the moment-strain curves of Fig. 5 and similar sets for the other shapes and for strain-hardening materials, the variation of stress, strain, and bending moment may be analyzed under various fatigue loading conditions.

A study of Fig. 2 readily reveals the behavior to be expected of stress for strain-softening materials under imposed constant-strain amplitude. The stress will decrease gradually up to 1000 cycles, beyond which it will rise slowly. Since the stress depends only on the strain for a given number of cycles, it will follow the same pattern for any cross-sectional shape. In the case of a strain-hardening material, the stress will gradually increase with number of cycles.

The behavior of the bending moment under constant-strain amplitude is shown in Fig. 6. Each curve represents the moment variation for a particular shape of section which is indicated by the symbol. The dashed curves represent the limiting case. An idealized shape having its entire area at the outer surface would undergo a change in moment equal to the change in stress. This relative change in force would occur in axial-stress tests under constant strain.

Here, for the first time, the effect of specimen shape is evident. Since the greatest change in stress occurs in the outer fibers, one would expect a specimen with the largest percentage of area away from the center to be most sensitive to the changing modulus. This fact is borne out in the figure where the "I" section has a much larger change in moment than the diamond section at the opposite end of the range.

The variation of strain and stress during constant moment tests is shown in Fig. 7. The effect of cross-sectional shape is again evident, as the change in either stress or strain under constant-moment loading is dependent on the specimen shape. To avoid confusion, the curves for the solid circular, square, and hollow circular shapes are omitted. They fall between the diamond and "I" shapes in the order shown in Fig. 6. This influence of shape on the stress-strain behavior will be discussed later.

The behavior of the strain and bending moment under constant-stress amplitude may be studied in the same manner as for the two preceding cases. To conserve space this analysis is omitted here; how-

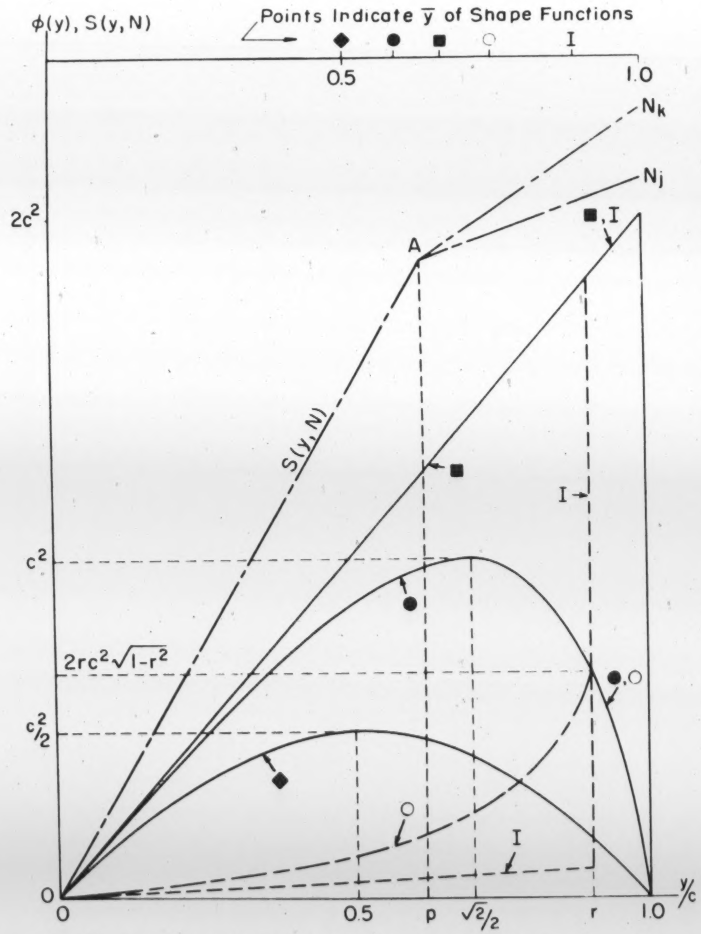


FIG. 8.—Graphs of the Shape Functions for the Five Specimen Shapes Studied and of $S(y, N)$.

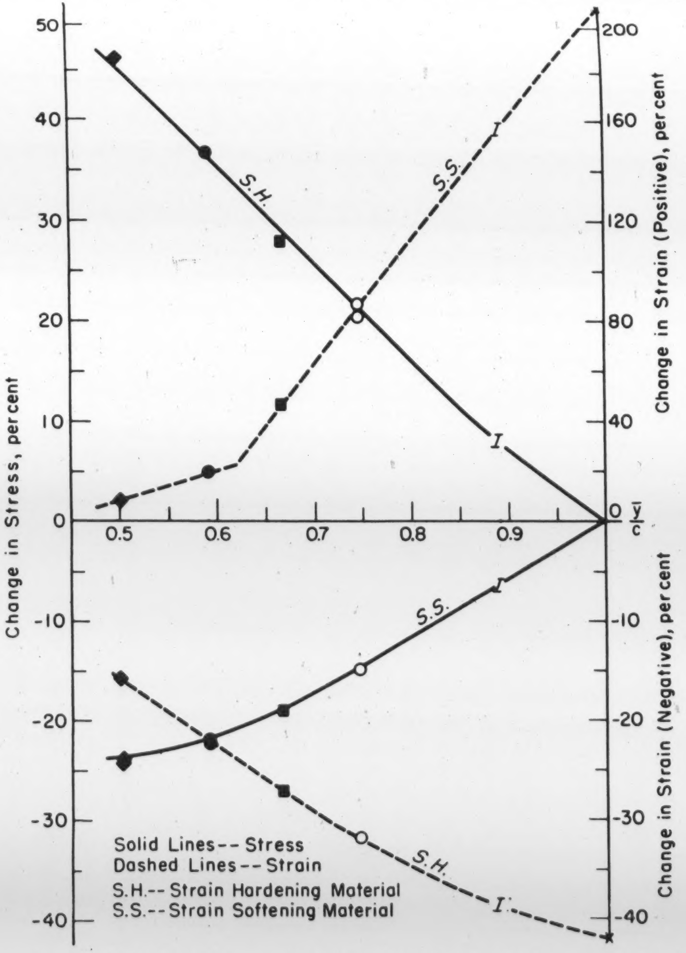


FIG. 9.—Relations Between the Per Cent Change in Stress and Strain Under Constant Moment and Distance to the Centroid of the Shape Function Area.

ever, a complete discussion may be found in reference (8). It should be noted, however, that the shape of the specimen does not affect the strain variations in this case just as it does not influence the stress behavior under constant-strain conditions.

EFFECT OF CROSS-SECTIONAL SHAPE ON FATIGUE BEHAVIOR

The sensitivity of the different shapes to the changing cyclic secant modulus, as noted previously, is felt only in the constant-moment test. In either the constant strain or stress test, the control is placed on the variable at the outer surface, and the shape of the section has nothing to do with its variation with repeated stress cycles. If the moment is controlled by external loading conditions, however, the stress distribution is involved, and hence the shape of the cross-section is a significant factor.

It is desirable to obtain a criterion for determining the relative amount that one shape will be affected by the changing modulus compared to another. In the following discussion, such a criterion will be developed.

The integrand of Eq 6, which is repeated here for convenience, is the product of two functions of y:

$$M = 2 \int_0^c S(y)\phi(y) \, dy \ldots\ldots (6)$$

The function ϕ was designated the "shape function" because it depends upon the shape of the section, but, more important, it determines how critically a given shape will be affected by the changing cyclic secant modulus.

Figure 8 shows the function S together with the ϕ functions for each of the five shapes. The long-dashed line OA together with the two lines AN_j and AN_k is a representation of the distribution of stress across one half of a section after two different numbers of cycles, N_j and N_k, the strain in the outer fibers remaining constant.[5]

Now the moment, for a given stress distribution, depends upon the character and magnitude of the function ϕ. However, for constant-strain tests the change in the moment with N depends only on that part of the function beyond the proportional limit of the stress curve (to the right of point p). The change will be greater if the value of ϕ in this range is greater. In particular, the per cent change will be higher if the ϕ curve in this plastic region is relatively higher than in the elastic range, and especially so if it is highest at the outer surface where the stress change is greatest. This observation provides a clue to the characteristic of the ϕ function that determines which shape is most sensitive to the changing modulus under constant strain.

The shape which theoretically would feel the change most would be one for which ϕ is zero all the way out to the surface and which has a finite value at the surface.[6] This shape is physically impossible, of course, but a shape whose ϕ curve is quite low from zero out to a point near the surface and then makes an abrupt rise would approach this idealized extreme case. The "I" section does just this, as can be seen in the figure. The diamond section, on the other hand, has the smallest portion of its ϕ area (the area under the ϕ curve) in the plastic region, and hence would be least sensitive to the change.

A quantity expressing the relative magnitude of the function in the outer region of the specimen is the distance from the center of the specimen to the centroid of the area between the ϕ curve and y axis. It should be noted that this is not necessarily the shape whose ϕ area has a larger moment with respect to the neutral axis. The relative moment would determine which shape has a larger absolute change in bending moment. A glance at Fig. 8 reveals that the square section obviously feels the greatest change in bending moment.

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[5] The illustration is for a strain-softening material. If the material were a strain-hardening type, N_k would be above N_j, but the analysis that follows is valid for either case.

[6] This limit condition for moment change under constant strain is analogous to the force change in an axial-stress test in which the strain is held constant.

In terms of relative change in bending moment with N, the distance to the centroid of the ϕ area is a useful and distinguishing criterion. Likewise, this quantity indicates which shape will feel the greatest change in stress and strain under controlled moment. This statement may be verified by reference to Fig. 9. In this graph the per cent change in strain and in stress under constant moment is plotted against $\bar{y}$, the distance to the centroid of the ϕ area. The points were obtained from the appropriate curve giving stress, strain, or moment as a function of N by noting the change in the selected variable at 10^4 cycles for each of the section shapes. These values were plotted against $\bar{y}$ for the particular shape.

The points for the idealized maximum-sensitivity shape are identified by an asterisk and located at $\bar{y} = 1.0$. The per cent change in strain under constant moment for this shape is equal to the per cent change in strain under constant stress. The change in stress under constant moment is, of course, zero for the idealized shape. Distances to the centroid of the various areas are indicated at the top of Fig. 8.

It may be observed that all of the stress curves converge at zero for the idealized shape for which $\bar{y}$ is 1.0. This is the expected behavior, since under constant moment there would be no change in stress in the idealized-shape specimen. No significance can be attached to the zero intersection of the strain curves, as this condition applies to a shape for which ϕ is identically zero in the plastic range. As long as this condition is satisfied, there can be infinitely many ϕ functions, in which $\bar{y}$ can have any value from 0 to pc, for which there would be no change in strain under constant moment. Actually, ϕ cannot be zero in the plastic range, and consequently the zero points of the strain curves have no practical significance.

The figure summarizes the shape effect, as discussed previously, in a compact manner. For a strain hardening material, the higher the ratio of $\bar{y}$ to c, the longer will be the fatigue life in a controlled-moment test, because the stress increase is less and the strain decrease is greater. Just the opposite relationship holds for a strain-softening material. The higher the value of $\bar{y}/c$, the lower will be the fatigue strength, since the strain increase is greater and the stress decrease less.

Another valuable application of the relationships in Fig. 9 is that of predicting for any given shape its fatigue sensitivity compared to that of a reference shape whose fatigue characteristics are known. For any shape of cross-section the ϕ function may be plotted and the centroid of the ϕ area located (graphically if analytical solution is not practicable). The relative position of $\bar{y}/c$ of the given shape on the curves of Fig. 9 determines whether the fatigue strength will be higher or lower than that of the reference shape. No numerical fatigue prediction may be made, of course, since as yet there is no satisfactory method of quantitatively evaluating the effect of varying stress on fatigue life.

EXPERIMENTAL PROGRAM

The foregoing analysis based on hypothetical material properties was intended to provide a means of studying the effect of varying stress-strain properties on fatigue strength without involving additional variables. The validity of this analysis was partially confirmed by experimental work which will now be presented.

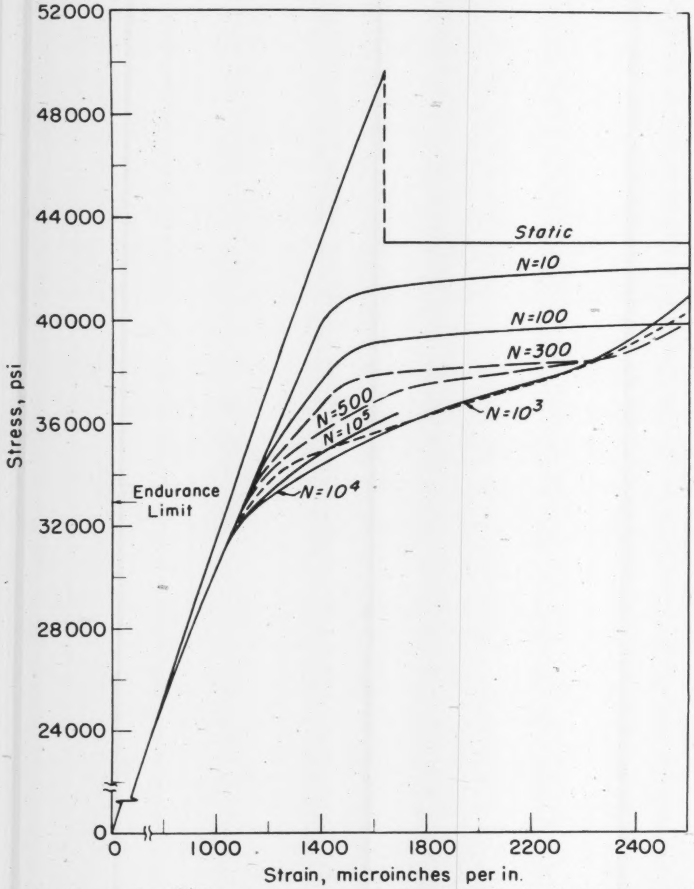

FIG. 10.—Cyclic Stress-Strain Locus Curves for 1020 Steel Under Axial Stress (Lidstrom and Lazan).

Fatigue Testing Machine:

Tests were conducted in a machine developed specifically for this work. The specimen is subjected to reversed-flexure by means of a controlled deflection arm whose amplitude may be varied at will without stopping the test. It is thus possible to impose any desired fatigue loading conditions on the specimen. Instruments for measuring and autographically recording the bending moment and strain in the specimen permitted continuous recording of these variables during the test. A detailed description of the equipment is given in reference (**8**).

Material:

The test program was performed on normalized SAE 1020 steel described in reference (8). In Fig. 10, cyclic stress-strain locus curves of mild steel are shown. These curves, obtained in prior work from axial-stress tests (6), were developed, with the exception of the static curve, from data from several tests. The strain amplitude at various numbers of cycles was recorded, and from these data points were plotted at the stress level of the test. Each of the locus curves shown in Fig. 10 connects all of the points for the indicated number of cycles. The strain-softening characteristic is indicated by the lowering of the stress-strain curve with increasing number of cycles. After 10^4 cycles, slight strain hardening begins in this material, as indicated by the somewhat higher location of the 10^5 cycle curve. The stress at a strain of about 2700 microinches per in. remains reasonably constant with cyclic stress history, and thus the region of greatest change is in the vicinity of the yield point.

Test Specimens:

Two shapes of specimen cross-section were employed. These shapes were so chosen that they provide an appreciable difference in response to the varying modulus and that nearly identical machining processes might be employed. The two types of specimens are hollow and solid circular in cross-section having a longitudinal straight taper. Further details of specimens are given in reference (8).

Test Procedures:

At the start of the test, the desired strain or moment was obtained by adjusting the stroke. After starting the test, the stroke was readjusted according to a precalculated pattern in order to produce the prescribed test conditions. Because of the rapid changes in cyclic secant modulus in the early stages of a test, the machine was stopped at the neutral position after the completion of each cycle. During each individual cycle, however, the machine was rotated by the motor at the usual speed of 15 cpm. In these early cycles, a moment-strain hysteresis loop was recorded for each cycle. After about 10 cycles, the machine was allowed to run continuously, and the data were recorded at increasing intervals, the stroke being adjusted as necessary while the machine was running.

Moment-Strain Relationships:

To compare the theoretical moment-strain relationship with experimental results, computations were made using the actual cyclic stress-strain locus curves of Fig. 10. Using Eq 3, sets of moment-strain values were computed by a numerical integration process.[7] By repeating the process for different values of N, a family of moment-strain curves similar to those of Fig. 5 were obtained for both solid and hollow circular specimens. From these curves, the moment-strain-stress behavior during given fatigue tests can be predicted. These predictions are presented below in connection with the experimental results.

RESULTS AND DISCUSSION

Three series of bending fatigue tests were conducted on both solid and hollow specimens of 1020 steel. In the first series, the strain amplitude in the outer fibers was held constant throughout the test. The second series consisted of constant-stress amplitude tests, while the moment amplitude was maintained constant in the third set of tests.

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[7] This procedure produced more accurate results than could be obtained by approximating the locus curves by two straight-line segments. Although these locus curves differ from the hypothetical curves in this respect, the results of the hypothetical analysis portray the same general trends.

Tests Under Constant Strain Amplitude:

In this series of tests the strain amplitude was maintained constant throughout the test by adjusting the stroke as necessary. Although the total deformation of the bending arm, grips, and specimen remains constant under constant stroke, that portion occurring in the gage length of the specimen increases as the modulus decreases. A constant-deflection test therefore does not necessarily maintain constant strain in the specimen, although the change may be relatively small if the specimen stiffness is much less than that of the loading system.

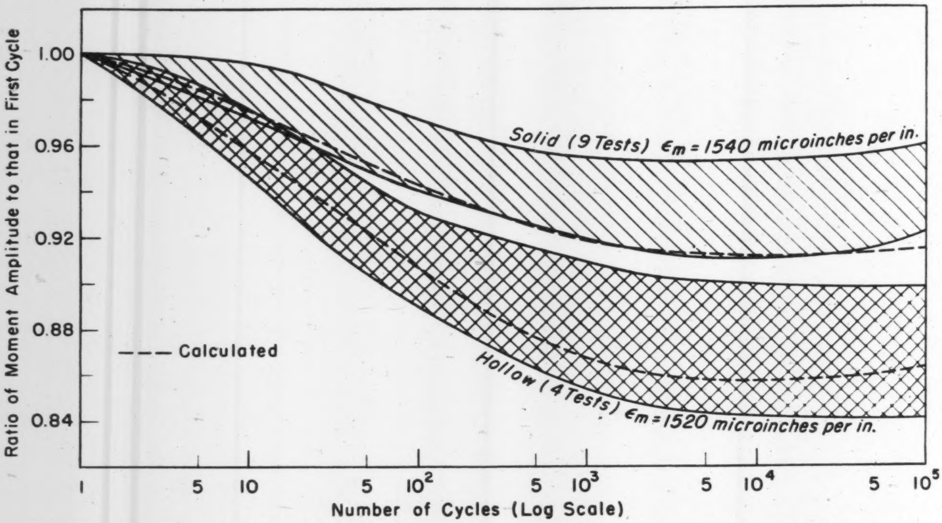

FIG. 11.—Moment Variation for Solid and Hollow Specimens for 1020 Steel Under Constant Strain, Showing Scatter Bands.

It is apparent from a study of Fig. 10 that if the strain amplitude is held constant, the stress will decrease significantly from its initial value. A direct experimental verification of this stress behavior is of course impossible with techniques available at the present time. However, an indication of the stress redistribution may be gained by measuring the bending moment.

Figure 11 shows the variation of bending moment with number of cycles for both hollow and solid specimens. In this graph, the scatter bands are shown in order that the amount of dispersion may be observed. The band for solid specimens, which includes the curves from nine tests, has a maximum width of about 4 per cent as compared to a 7 per cent maximum change in moment at the center of the band.

For the hollow specimens, the change in moment is about 13 to 15 per cent, which is just about double that of the solid specimens. The agreement between calculated values, as indicated by the dashed curve, and the experimental values appears to be quite good.

Constant Stress Amplitude:

Since there is no reliable experimental method of measuring stress in a metallic specimen, the best substitute is to measure the strain at the surface, and if the stress-strain relationship is known, the stress may be calculated. In the constant-

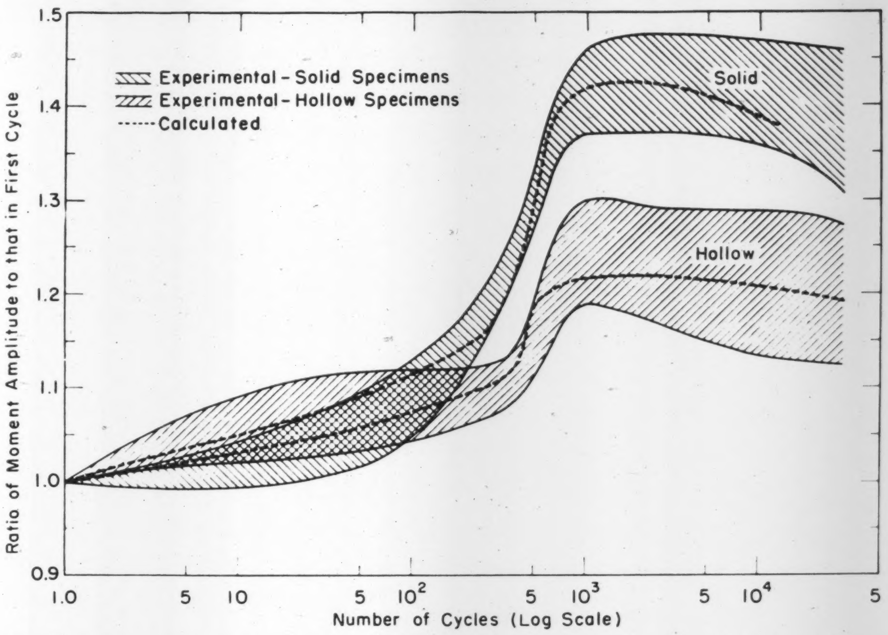

Fig. 12.—Moment Variations for Hollow and Solid Specimens of 1020 Steel Under Constant Stress, Showing Scatter Bands.

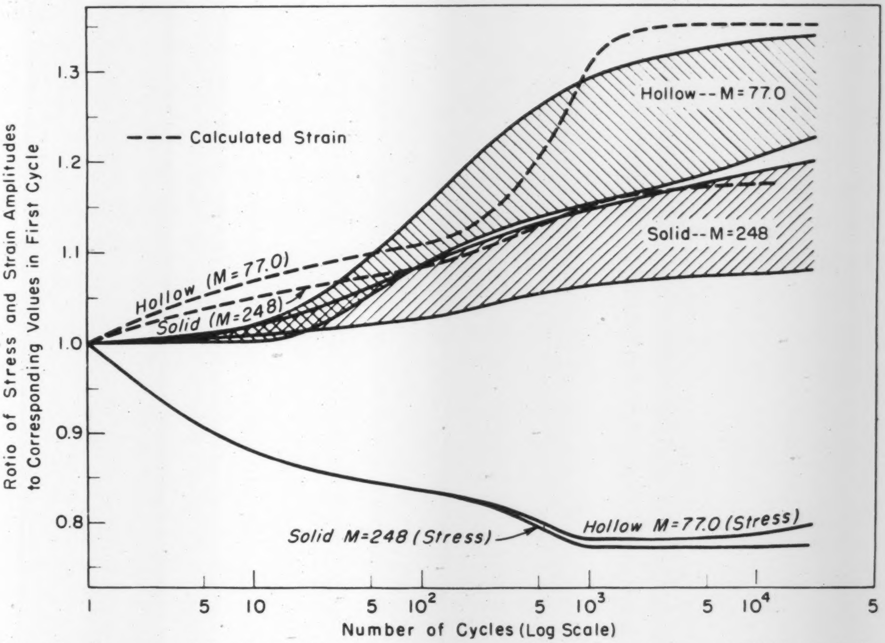

Fig. 13.—Stress and Strain Variations for Hollow and Solid Specimens of Mild Steel Under Constant Moment Amplitude.

stress tests, this procedure was employed, the cyclic stress-strain locus curves of Fig. 10 providing the relation of stress to strain. The intersections of a horizontal line at the selected stress level with the individual curves of Fig. 10 give the value of strain for the respective number of cycles. These sets of values of ϵ_m and N may be used to construct a curve showing the variation of strain during a test under constant stress amplitude. The strain amplitude variation indicated by such a curve was imposed on the specimen during the tests.

TABLE I.—SUMMARY OF FATIGUE RESULTS.

Type of Test	Number of Tests	Cycles to Failure, avg.	Standard Deviation	Initial Strain Amplitude, microinch per in.
SOLID SPECIMENS				
Constant strain	12	320 000	81 500	1540
Constant moment	6	120 000	31 600	1540
Constant stress	4	55 000	3 900	1200
HOLLOW SPECIMENS				
Constant strain	4	115 000	46 800	1510
Constant moment	3	30 000	6 300	1540
Constant stress	4	20 000	3 400	1200

An experimental indication of the stress distribution and its variation was obtained by measuring the bending moment at frequent intervals throughout each test. Figure 12 is a plot of the moment variation with N. Again, the scatter bands are shown in order to display the dispersion of results due to variability in material properties. This factor will be significant in the discussion of fatigue results later. The fact that the calculated curves (dashed) are within the scatter bands over the entire life indicates that the stress was close to the desired level throughout the tests.

Constant Moment Amplitude:

In the first cycle of these tests, a strain of 1540 microinches per in. (the strain amplitude maintained in most of the constant-strain tests) was imposed on the specimen. Because of variability in material properties and dimensions of the specimens, this procedure did not impose the same moment on each specimen. However, because the same initial strain was developed in each case, it is felt that this common initial condition provides a sound basis for comparing fatigue strengths under the various test conditions.

In Fig. 13 the behavior of strain and stress during the fatigue tests under constant moment are shown. It is apparent, even with the amount of scatter present, that the strain increase in the hollow specimens is significantly higher than in the solid ones. In the case of the solid specimens, the calculated curve (dashed) falls along the upper border of the experimental band. In the case of the hollow specimens the discrepancy appears more serious. Reasons for these differences are discussed in some detail in reference (8).

The stress curves show a considerable decrease from the initial value, with the solid specimens having a slightly greater decrease. It should be noted, however, that these stress curves are determined from calculations under the assumption that the cyclic locus curves of Fig. 10

represent the stress-strain relationship in each specimen. The dispersion in strain values noted above indicates that the stress-strain relationship may vary somewhat. Nevertheless, these curves, based on the average strain values, represent the stress trends though the degree of change may be questionable.

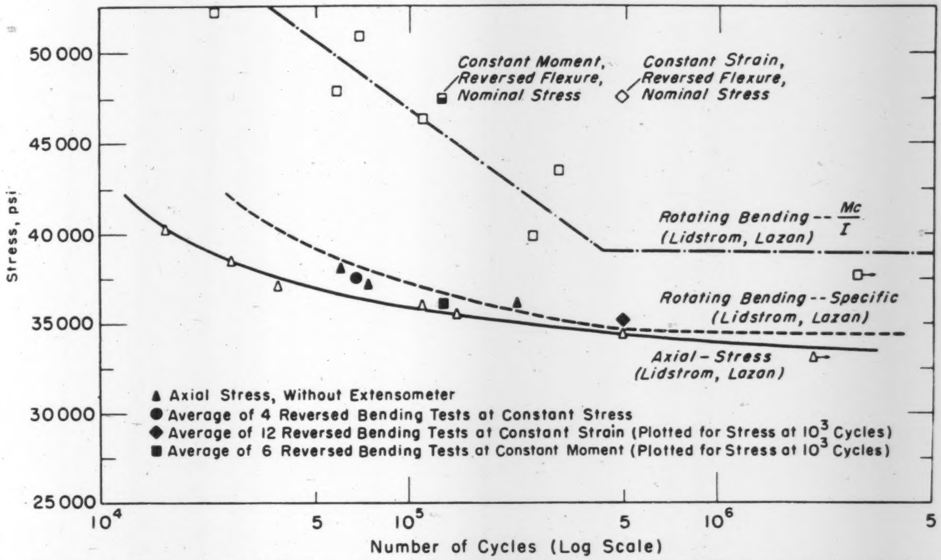

FIG. 14.—Comparison of Fatigue Strength of Solid Specimens of 1020 Steel Under Various Conditions.

Comparisons Between the Three Loading Conditions:

A summary of the variations of strain-stress, and moment with N under various loading conditions may be obtained from Figs. 10 to 13. A comparison of the strain curves for solid specimens reveals that under constant moment the strain has the same initial value as maintained in the constant-strain tests, but rises about 12 per cent above that value during the test. In the constant-stress test the initial strain is 22 per cent lower, but it rises to a value 45 per cent higher than the constant-strain level. This comparison of strain behavior indicates that the longest life will be found under constant-strain conditions; the constant-moment tests will give intermediate life, while the shortest life will be found under constant-stress tests. A similar comparison of stress curves substantiates this statement. The curves for hollow specimens indicate the same trends, the only difference being in the degree of the changes.

Fatigue Results:

Table I gives a summary of all fatigue results on mild steel. The table is divided into two principal groups separating solid and hollow specimens. For a given specimen type, it is quite evident that an appreciable difference in fatigue life results from the three types of tests, in which the initial conditions are identical. The constant strain test shows the longest life, while the constant-moment value is less. The constant-stress test gives the lowest result even at a level in which the initial strain is less than for the other two cases. The same relative position is evident for both types of specimens.

The evident difference in fatigue life of the solid and hollow specimens in the constant strain and stress tests is apparently the result of factors other than the changing cyclic modulus. A study of these factors is beyond the scope of this paper. A difference between solid and hollow specimens under constant moment, however, is expected from the analysis of changing modulus.

Comparisons with Prior Results:

In Fig. 14 are shown the *S-N* curves obtained on the same lot of steel as was tested in this project (6). The solid curve is the result of direct-stress tests, while the dashed curve is from rotating-bending data. The short-dash curve is from rotating-beam data corrected to show specific stress values (at 10^3 cycles). The plotted points are from data obtained in this investigation, their source being indicated by the code.

The direct-stress curve was obtained in tests in which strain was measured by an extensometer attached to the specimen. Its knife edges marred the surface slightly, and all failures occurred at these points. Three tests (shown in the figure by the solid triangular points) have since been run without an extensometer, and all three yielded slightly longer life than those which established the curve. This indicates that the true direct-stress curve should perhaps be farther to the right than shown by the solid line.

The average life of four solid specimens in bending under constant stress are shown by the circular point. The solid square point indicates the average life of six solid specimens tested at constant-moment. These tests were not run at a constant stress, but for comparison purposes their stress at 1000 cycles (after which relatively little change occurs) was used in plotting the point.

The nominal (MC/I) stress for this set of tests, indicated by the half solid square point, falls quite close to the nominal rotating-bending curve. The solid diamond point is for the average of 12 reversed-flexure tests at constant strain, plotted for the stress value at 10^3 cycles. At the same life but considerably higher is the corresponding nominal stress point (hollow diamond).

Obviously, the nominal-stress values for the three tests (rotating bending, reversed flexure at constant moment, and reversed flexure at constant strain) are unreliable. However, the points for axial stress (without extensometer) and for reversed flexure under constant stress, constant moment, and constant strain fall in a consistent curve which agrees very well with the corrected rotating-bending curve. Thus there appears to be little difference between the results of all of these tests if the actual stress is used.

SUMMARY AND CONCLUSIONS

By means of hypothetical stress-strain locus curves, an analysis was made of the effect of changing cyclic properties on stress redistribution and its influence on the fatigue life to be expected for various cross-sectional shapes under different loading conditions. Curves consisting of two straight-line segments were assumed to represent the cyclic stress-strain properties of the material. The proportional limit of the material remains fixed and the slope of the upper (plastic) leg of the stress-strain locus curve varies with cycles of repeated stress. Using these locus curves, expressions for the bending moment were obtained for each of five shapes of cross-section: (*a*) square, (*b*) diamond, (*c*) circular, (*d*) hollow circular, and (*e*) "I" section. From these moment equations, two sets of moment-strain curves were obtained: one for a strain-hardening material, and the other representing a strain-softening material. These families of curves together with the cyclic locus curves form the basis for analyzing the behavior of the moment, strain, and stress under various conditions during fatigue tests.

Three different types of reversed-flexure tests were performed on solid and hollow cylindrical specimens of mild steel, the desired test conditions being produced by varying the deflection at the end of the bending arm as required during the tests. In the first type of test, the strain amplitude was held constant. In the second type, constant stress amplitude was maintained by varying the strain in a predetermined manner. The third type of test was performed under constant-moment amplitude. In all of the tests, the bending moment at the center of the specimen and the strain in the outer fibers were simultaneously recorded at frequent intervals.

A check on the assumptions and hypothesis regarding the effects of a changing cyclic secant modulus was accomplished by comparing calculated with experimental curves of the bending moment and strain. Using a numerical integration process, the bending moment for a given maximum strain was computed from cyclic locus curves previously determined in direct stress. In this manner, a family of moment-strain curves was developed for each of the two specimen types similar to those obtained in the hypothetical analysis. These curves provided a means of predicting the moment and strain variation throughout the test.

The results of the constant-strain tests showed a decreasing bending moment in the early stages for both solid and hollow specimens. The hollow specimens experienced a greater percentage reduction in moment, as was predicted by the theory. The experimental curves followed the calculated ones quite closely for both specimen types, thus indicating the validity of the assumptions and the stress distribution calculated therefrom.

In the constant-stress tests, the bending moment increased for both the hollow and solid specimen, the solid type showing the larger percentage change. Again the calculated curves were in good agreement with the average of the experimental work.

The results of the constant-moment tests revealed trends predicted by the theory, the stress decreasing and the strain increasing for both types of specimens. The scatter bands for the experimental strain curves followed the trends predicted by the calculated curves. The increase in strain for the hollow specimens was higher than that of the solid ones, and consequently the stress decrease was less.

The fatigue life determined by the three types of test was quite different, as was anticipated from a study of the stress behavior. For both types of specimen, the constant-strain tests yielded the longest life, the constant-moment tests were intermediate, and the constant-stress tests gave lowest strength values, even though the initial strain level was below that of the constant-strain and moment tests.

The following conclusions may be drawn from the results of this investigation:

1. The agreement between the calculated variation of strain and bending moment and the measured values demonstrates the validity of the assumption that individual filaments of the material behave the same under bending conditions as they do under axial loading.

2. The distribution of bending stress during fatigue tests changes with cyclic stress history as a result of the change in

the cyclic secant modulus. Furthermore, the degree of change depends upon the shape of the cross-section. In general, a shape having a greater portion of its area remote from the neutral axis is more sensitive to the change than one having the bulk of its area near the axis. These observations were confirmed by the experimental results on mild steel.

3. Stress redistribution resulting from the changing cycle modulus affects the fatigue life. Though the initial strain amplitude be the same, the fatigue life of a strain-softening material increases with testing conditions in the following order:

(*a*) Constant stress amplitude,
(*b*) Constant moment amplitude; and
(*c*) Constant strain amplitude.

For strain-hardening materials, the order is reversed.

4. In the case of mild steel, there appears to be no appreciable difference in fatigue strength among any type of reversed flexure test, rotating bending test, or even axial-stress test, providing the load is adjusted so that the same actual stress exists at 1000 cycles. From that point on, little change occurs in the cyclic modulus until cracks become large enough to affect the properties.

Acknowledgment:

The cooperation and encouragement of the Materials Laboratory, Wright Air Development Center, sponsors of this work, are greatly appreciated.

The able assistance of Mr. Byron K. Olson in performing the computations, of Miss Helen Thomas in preparing the figures, and of Mr. Fred E. Abigt in conducting the tests is gratefully acknowledged. The cooperation of Miss Audrey Vinck in typing the manuscript is appreciated.

APPENDIX

DEFINITIONS OF SYMBOLS AND TERMS

c—distance from the neutral axis to the extreme surface.
E—modulus of elasticity.
E_t—tangent modulus—slope of hypothetical stress-strain curve beyond proportional limit.
I—moment of inertia of a cross section with respect to the neutral axis.
k—ratio of wall thickness to outer radius of hollow specimens ($= 1 - r$).
M—maximum bending moment in a cycle at the center of the specimen.
M_1—maximum bending moment during first cycle.
N—number of repeated cycles.
p—ϵ_p/ϵ_m—ratio of strain at proportional limit to maximum strain.
r—ratio of inner to outer radii of hollow specimens.
S—maximum stress in a cycle.
y—distance from neutral axis (variable).
$\bar{y}$—distance from neutral axis to the centroid of the area under the curve of $\phi(y)$.
ϵ_m—maximum strain during a cycle in the outer fibers.
ϵ_p—strain corresponding to the proportional limit.
ϵ_1—strain amplitude in first cycle.
ϕ—shape function—that factor of the moment integral dependent upon shape of specimen cross section.
Cyclic Secant Modulus—the ratio of the maximum stress to the maximum strain of the cyclic stress-strain hysteresis loop. These maxima need not occur simultaneously.
Cyclic Stress-Strain Locus Curve—the locus of stress-strain points for which the stress value is the product of the strain and the corresponding cyclic secant modulus. (If the maximum stress and strain occur at the same point, this curve is the locus of maximum points of the hysteresis loops.)

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(3) B. J. Lazan and L. J. Demer, "Damping, Elasticity, and Fatigue Properties of Temperature-Resistant Materials," *Proceedings*, Am. Soc. Testing Mats., Vol. 51, p. 611 (1951).
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DISCUSSION

Mr. G. R. Gohn[1] (*presented in written form*).—The authors have done an excellent piece of work in furthering our knowledge of the factors that affect the fatigue strength of a given material.

On the basis of their theoretical studies, which they have confirmed by well-planned laboratory tests, it has been demonstrated once again that useful fatigue data cannot be obtained from the simple rotating beam, reversed flexural or repeated axial load fatigue tests without further consideration of the many factors which may operate to alter the stress-cycle relationship.

For many years, factors such as size, shape, surface condition, stress concentration, residual stresses, quality of the material, and environment have been studied to evaluate their effect on fatigue life. More recently, grain size, test temperature, stress distribution, range of stress, ratio of stress range to mean stress and spectrum types of loading have been shown to markedly affect the results obtained in conventional fatigue tests. The importance of cycle frequency and variations in prior stress history have also been demonstrated by recent experiments. To these must now be added the effect caused by variations in stress distribution which have been shown by the authors to occur during the course of the fatigue test.

The magnitude of the effect caused by changing modulus has been theoretically calculated by Messrs. Blatherwick and Lazan. The reversed flexure tests which the authors have reported for mild steel—and the more limited data on aluminum alloy 2024-T4—confirm their assumptions. To their experimental data, I should like to add the results of two series of tests that we have made which likewise demonstrate that changing cyclic properties do alter the stress distribution during fatigue tests. The first of these tests were reported in 1951 by Gohn and Ellis and are shown in Figs. 12 and 13 of their paper which was presented before this Society.[2] The studies involved determination of the change in bending moments for cantilever beam specimens of a high-purity lead extruded as pipe and a lead-antimony-zinc alloy cable sheath, both of which were tested in the form of flattened strip. The changes in bending moment were measured during repeated bending tests at $\frac{1}{4}$ cpm and at constant deflection. Strain measurements made with SR-4 gages during the course of the cyclic tests indicated no significant change in strain during the fatigue tests. Instantaneous bending moments, determined on the Boettler and Werring pendulum tester[3] accompanying Fig. 15 showed measurable changes with repeated cycling. The magnitude of these changes is shown in Figs. 16(*a*) and (*b*) in which the data are replotted in the manner used by the authors.

Similar changes in instantaneous bending moments occurred during reversed bending fatigue tests at a constant deflection of ±0.750 in. on three grades of extruded aluminum strip when tested in two different conditions and at two different speeds $\frac{1}{4}$ and 18.5 cpm. The data from these studies are shown in Figs. 17(*a*) and (*b*). It should be observed that the data show that cyclic stressing has different effects on the bending moments of the several materials depending upon the strain range, the condition of the material, and the frequency of cycling. Other factors not covered by our studies may also have an effect. A study of the authors' work and the additional data offered in this discussion in the light of dislocation theory should be most interesting.

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[1] Member of Technical Staff, Bell Telephone Laboratories, Inc., New York, N. Y.

[2] G. R. Gohn and W. C. Ellis, "The Fatigue Test as Applied to Lead Cable Sheath," *Proceedings*, Am. Soc. Testing Mats., Vol. 51, pp. 721–740 (1951).

[3] U. S. Patent 2258276 of October 7 (1941).

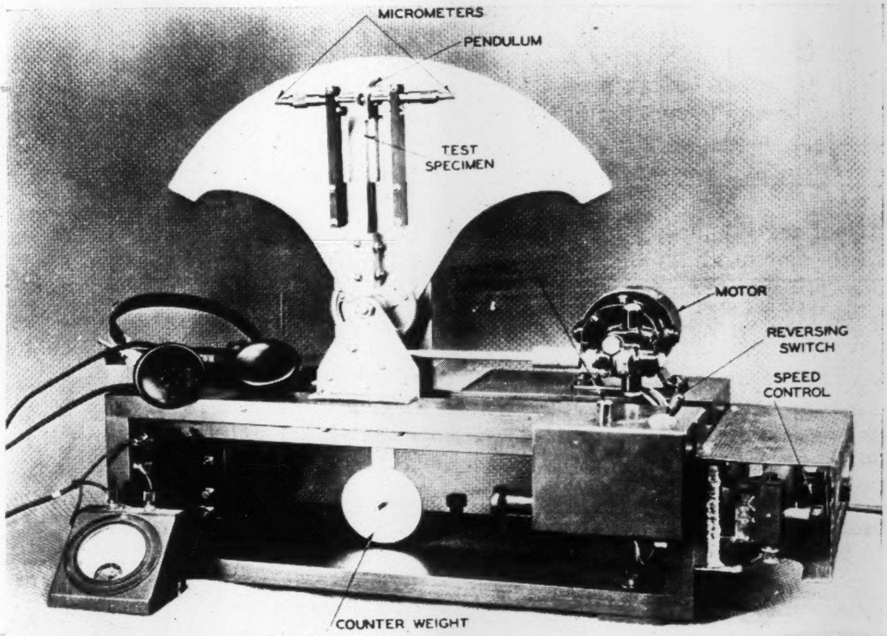
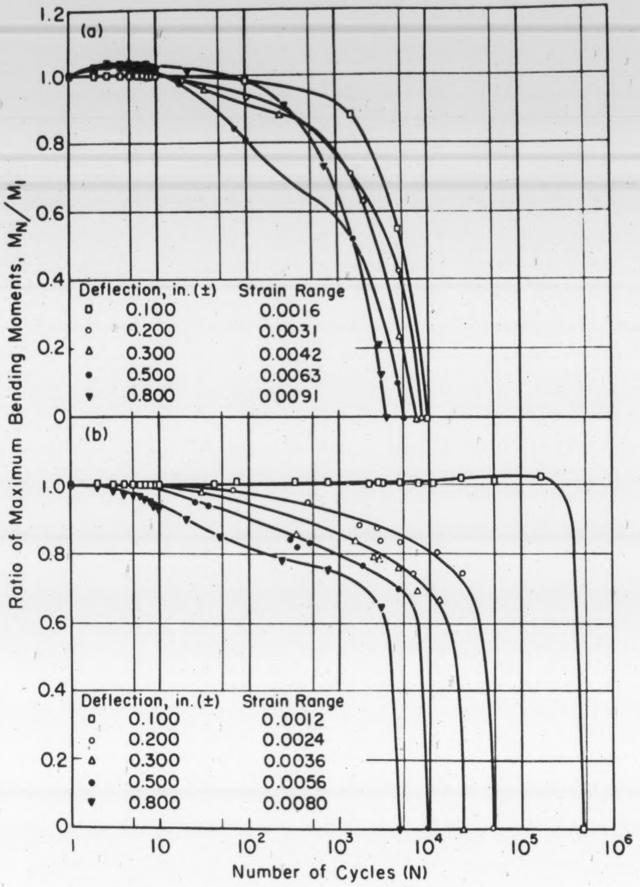


FIG. 15.—Bend Tester Used in Cylic Tests to Evaluate Instantaneous Bending Moments.

I should like to offer one suggestion and that pertains to the description of the mechanism as one of "strain hardening" or "strain softening." These terms have already been established in metallurgical practice to mean something slightly different from the phenomenon described by the authors. For example, all three of the grades of aluminum which we tested are capable of being "strain hardened" when cold worked even though, as shown by our data, they may show a loss in bending moment when subjected to cyclic straining at constant deflection. Obviously to classify these materials as "strain softening" is confusing. Some other term might be used to better describe this phenomenon.

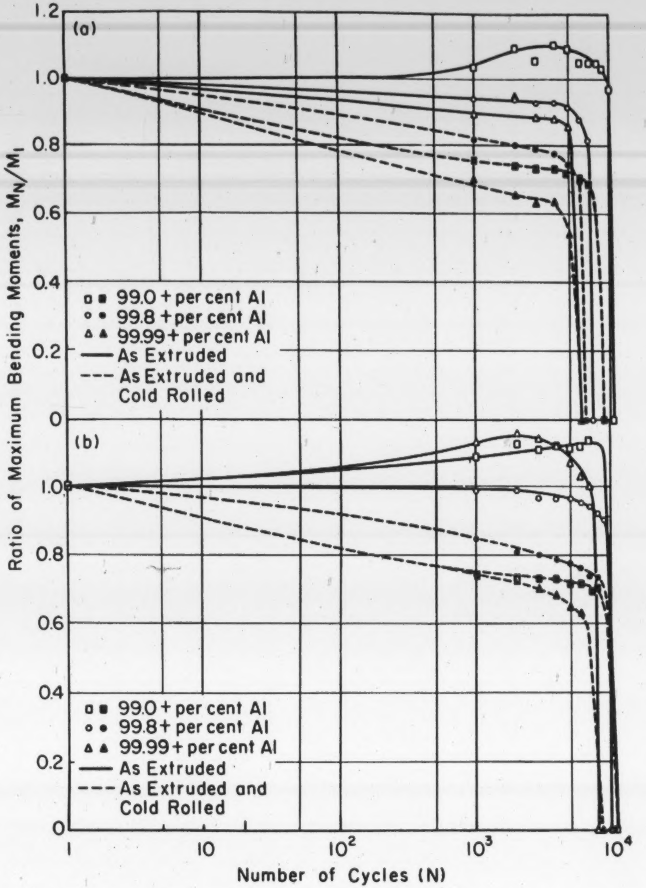
A MEMBER.—At what rate were the stress-strain curves obtained shown in Fig. 1 and Fig. 12?

Secondly, how did you maintain a constant stress during bending fatigue test? I think that was a bending fatigue test, in which you said you had a constant stress.



(*a*) High-Purity Lead (99.995 per cent lead) Pipe.
(*b*) Lead-0.65 per cent Antimony-0.25 per cent Zinc Sheath.
FIG. 16.—Changes in Maximum Bending Moment with Cyclic Straining.

Tested at ¼ cpm, 85 F and zero mean strain.



(*a*) Tested at ¼ cpm, 85 F, and zero mean strain.
(*b*) Tested at 18.5 cpm, 85 F, and zero mean strain. Deflection ± 0.750 in.
FIG. 17.—Changes in Maximum Bending Moment with Cyclic Straining—Extruded Aluminum Strip.

MR. BLATHERWICK (*author*).—The bending fatigue tests were reverse flexure. Both the axial tests, from which the cyclic modulus curves were obtained and the bending tests, in which the bending moment was measured, were run at a frequency of about 15 cpm. This frequency was used at all times when measurements were taken. Between measurements, the speed was increased to as much as 300 cpm.

The stress was maintained constant in the bending test by following a pattern of strain calculated from the cyclic locus curves. A horizontal line across the family of cyclic locus curves (Fig. 10) determined the strain value that should occur at the indicated number of cycles at the selected stress. Then, in the bending test, this strain pattern was followed by adjusting the stroke during the course of the test.

Admittedly, this procedure is based on the assumption that the cyclic locus curves hold for each specimen. Some disparity was observed in the material properties from specimen to specimen, as can be seen by the scatter bands in the results that were obtained.

In reply to Mr. Gohn's discussion, we should like to thank him for the interesting addition to the material that we presented. We agree with him wholeheartedly in regard to his criticism of the terminology "strain softening and strain hardening." We really should call it cyclic-strain hardening or cyclic-strain softening, or perhaps some better term, to distinguish it from the usual meaning of strain hardening and strain softening. The meaning that is intended in this paper is that under cyclic conditions the stress corresponding to a given strain decreases with number of cycles for a cyclic-strain softening material. If it is a cyclic-strain hardening material, the stress increases with number of cycles.

The variation in effects at different stress levels indicated in Fig. 16 of the discussion by Mr. Gohn is believed to be the result of variation in crack formation, as well as in strain rate. It has been observed by a number of investigators, among whom the work of Hunter and Fricke[4] is an example, that cracks start to form much earlier at higher stress levels and grow more rapidly. This effect is more pronounced in some materials than in others, and apparently lead and aluminum differ considerably in this respect.

We should point out further that the curves presented in this paper pertain to that portion of the fatigue life before cracks develop sufficiently to affect the bending moment. We observed cracks early in the tests, but these cracks did not become large enough to produce a measurable change in bending moment until about the last 5% of the fatigue life. In one test under constant strain amplitude, the bending moment was observed to drop off to zero as failure approached, similar to the curves of Figs. 16 and 17 of Mr. Gohn's discussion. In all other tests, we did not attempt to follow changes in bending moment during these final stages.

Further work by the authors is underway to investigate differences in cyclic modulus effects on fatigue for different materials.

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[4] M. S. Hunter and W. G. Fricke, "Metallographic Aspects of Fatigue Behavior of Aluminum," *Proceedings*, Am. Soc. Testing Mats., Vol. 54, p. 717 (1954).

FATIGUE CRACK PROPAGATION IN ALUMINUM ALLOYS*

By M. S. Hunter[1] and Wm. G. Fricke, Jr.[1]

Synopsis

Metallographic observation of fatigue cracks in aluminum alloys from their inception to final complete failure of the specimen has revealed the manner in which fatigue cracks form, propagate, and interact. It has been found that the length of a fatigue crack is related exponentially to the number of stress cycles and that the rate of propagation bears a similar relationship to the stress level. It has been observed that incipient fatigue cracks will propagate at stresses below the endurance limit and at rates which are simple extrapolations of those observed above the endurance limit.

It has been shown that fatigue cracks start to form very early in the life of a material and that a large portion of the fatigue life of a part is consumed in the growth of the initial fatigue crack. Increasing interest is being shown, consequently, in the problem of fatigue crack propagation. Little is known of the actual mechanism by which fatigue cracks propagate. There is, moreover, intense interest in the practical problem of determining the usefulness remaining in a structural part once a fatigue crack has formed. So little is actually known of fatigue crack propagation that there is no agreement in the literature on even so elementary a point as how the rate of growth of a crack changes as the crack becomes larger. Very little is known of how this rate of growth is affected by stress level.

For these reasons, it was decided to employ techniques which had proved capable of detecting fatigue cracks at an early stage in order to follow the development and progress of individual cracks and to study the processes by which they extended and reacted with one another. This paper describes the manner in which fatigue cracks propagate on a microscopic scale, some of the intricacies of crack propagation, and the relationships between crack length, number of cycles, and stress level.

Material and Testing Method

Most of the experimental observations of fatigue crack propagation were made on 6061-T6 aluminum alloy. Sufficient observations were made, however, on 2024-T3, 7075-T6, and X7375-T6 alloys to ensure that the principles were generally true of the structural aluminum alloys.

The 6061-T6 alloy was obtained as commercial $\frac{3}{4}$-in. diameter rod having a tensile strength of 44,700 psi, a yield strength (0.2 per cent offset) of 37,700 psi, and an elongation of 19.5 per cent over a 2-in. gage length. Smooth rotating-beam fatigue specimens having a minimum diameter of 0.300 in. were made from this material.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] Assistant Chief, Metallography Division and Research Metallurgist respectively, Alcoa Research Laboratories, Aluminum Company of America, New Kensington, Pa.

Before testing, each specimen was chemically polished to remove scratches and leave a metallographically polished surface. Testing was interrupted periodically in order to obtain plastic replicas of the specimen surface. These replicas reproduced the surface details faithfully and could be examined under the microscope at any magnification.

By comparing the series of replicas for any test, it was possible to follow the growth and interplay of all the fatigue cracks in a specimen. Direct measurements of crack length could be made from the replicas using a graduated micrometer eyepiece on the microscope. For convenience, however, the length of large cracks was determined from a tracing made from the replicas by a camera lucida arrangement using a magnification of about 50×. It was felt that the true measure of the length of a crack was the amount of material which it had traversed. Consequently, the length of large cracks was measured along the crack itself. For small cracks, this method was cumbersome and lengths were measured between crack ends in a direction perpendicular to the stress. Both methods of measurement gave similar results.

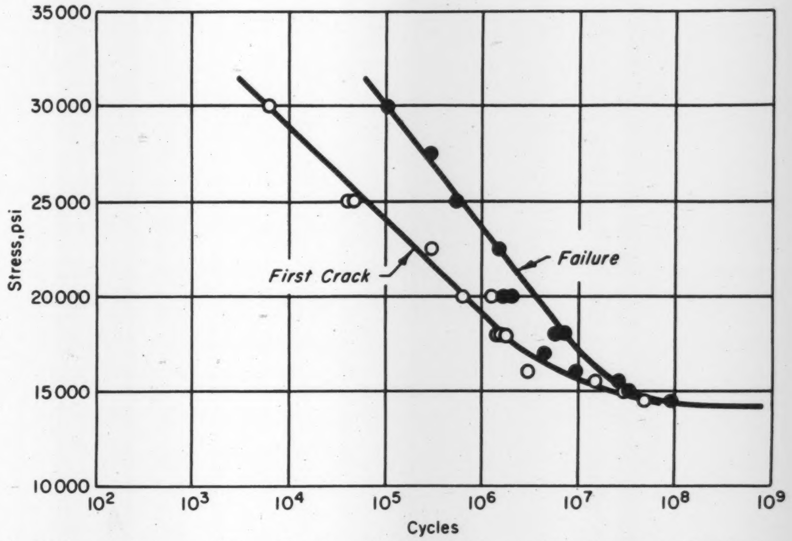

FIG. 1.—Relation Between First Crack and Failure Curves for 6061-T6 Aluminum Alloy.

TEST RESULTS

First-Crack Curve:

Before fatigue cracks can propagate, they must be initiated. When the number of cycles at which a microscopic size crack first appeared in each specimen was noted on the S-log N plot, the points for all tests were found to lie along a curve defining the first inception of cracking for the various stress levels (Fig. 1). The first-crack curve so plotted converges on the conventional failure curve at the low stress levels as it does in all structural aluminum alloys (**1**, **2**).[2] Scatter of points about the first crack curve is about the same as the scatter of points about the failure curve, indicating that much of the scatter in fatigue testing is the result of vagaries of crack initiation. In general, a specimen which cracked late, failed late, and the specimen which cracked first, failed first.

Slip which probably precedes cracking even in the structural alloys was not sufficiently pronounced to be resolvable by the optical microscope, except in regions of large stress concentration, such as at the ends of a large crack, between two close cracks, or in the fork of a crack. Consequently, "curves of progressive change" other than the "first-crack curve" were not plotted. Metallographic observation of the progress of failure consisted, then, of watching the formation and propagation of fatigue cracks. At low stresses, this comprised the watching of only the one dominant crack, but at high stresses, it necessitated following the progression and interplay of a large number of interrelated cracks.

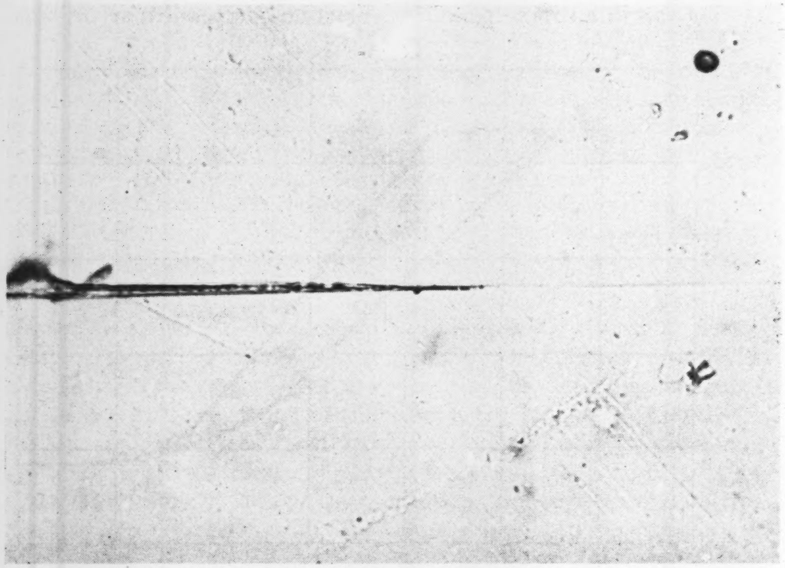

FIG. 2.—Unusually Long Plastically-Deformed Region at Tip of Fatigue Crack in X7375-T6 Aluminum Alloy. Plastic replica (×500).

Mechanisms of Crack Propagation:

Operating in advance of a fatigue crack is a stress field that amplifies the nominal stress many times and accelerates the sequence of events in regions before the crack. A large crack may be responsible for widespread deformation at its ends. Independent cracks tend to join because of their overlapping stress fields.

Largely because of the large stresses at the tip, a crack tends to grow in length as cycling continues. This extension may be accomplished by a number of modes: either by parting of the material at the crack tip, by formation of a branch a short distance behind the tip, or by formation of a new crack that unites with the original crack.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1046.

Extension of the crack tip probably occurs by a process closely akin to crack initiation, the stress concentration at the tip accelerating the passage of the material through the customary stages of fatigue. This is essentially the mechanism proposed by Head (3). Occasionally, a narrow, plastically-deformed region was observed at the end of a crack. This was the material that had not yet ruptured. Figure 2 shows such a region of unusual length.

Although the cracks propagated nominally in a direction perpendicular to the stress, each was composed of many jogs which lay at almost any angle to the stress. The end of the crack apparently did not move forward at a uniform rate but by a series of bounds. This was evidenced by the fact that when a crack was observed, the tip of the crack was very often just at the point where the crack eventually changed direction to form a jog.

When the end of the crack reached a region substantially stronger than the material through which it had been passing, it would often skirt the barrier by forming a branch crack a short distance from the tip. The "strong" region thus by-passed could have been an unfavorably oriented grain, or an area of unusually high solute concentration, or a cluster of constituents which blunted the stress field ahead of the crack. Once the branch crack had passed the original tip, the latter grew very little, if at all.

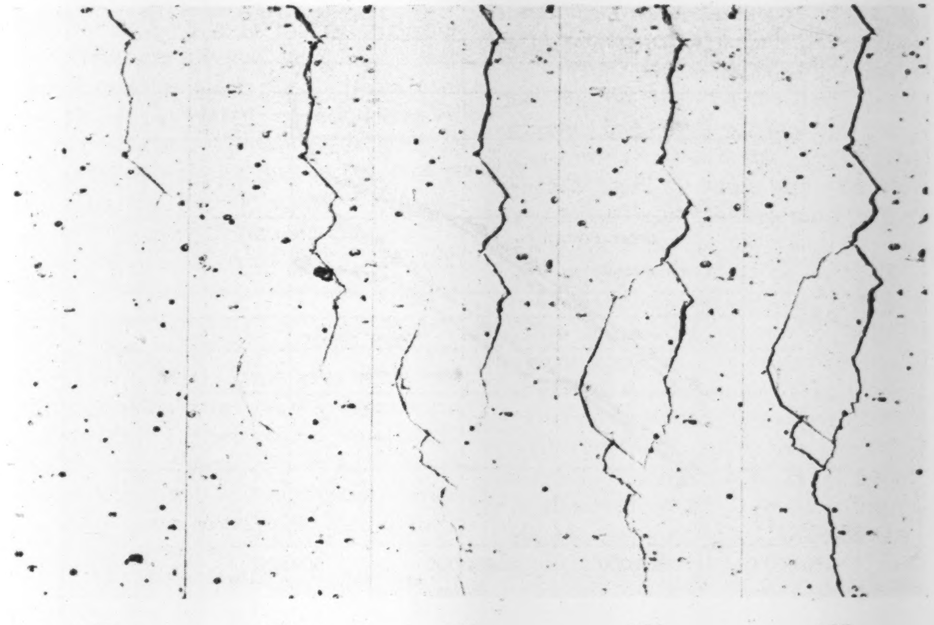

FIG. 3.—Growth of Fatigue Crack in 6061-T6 Aluminum Alloy Showing Formation of a Crack Ahead of the Main Crack as Well as the Branching Which Occurs Near a Crack Tip. Plastic replicas (×100). The numbers under photograph refer to number of Cycles in millions.

The branching near the end of a crack

must be viewed with some caution. A crack does not fork because it reaches two avenues of equal weakness, nor does it grow simultaneously along both branches. Conclusive evidence has been obtained that the large "branches" which sometimes appear in fatigue cracks at an advanced stage are the result of the joining up of two separate cracks. Since this joining is seldom end to end, a portion of one crack remains projecting from the composite crack in what appears to be a branch or fork.

There remains one other means by which cracks increase in length. Because of the stress concentrating effects operating at some distance in advance of the crack tip, cracks were often formed in areas that were originally just strong enough to resist the test load. The formation of cracks ahead of the main crack sometimes occurred even when the main crack was still advancing. The process was of more interest, however, when the tip of the main crack had become stopped or slowed. Many times, under these conditions, a small crack formed on the other side of the barrier and, under the onslaught of cracks from both sides, the barrier was broached. Figure 3 illustrates cracking ahead of a main crack. Although this process was not as important as simple branching at test stresses near the endurance limit, it assumed equal if not greater importance at higher stresses.

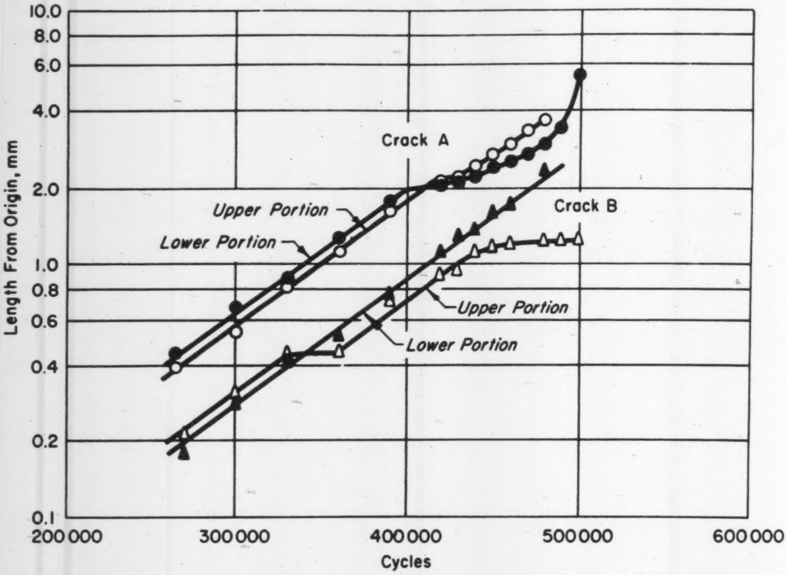

FIG. 4.—Interference of Growing Fatigue Cracks. Lower Portions of Crack *A* Came Abreast of Upper Portion of Crack *B* at 400,000 Cycles at Which Time These Ends Grew at Decreased Rates. Free ends continued growing unaffected. Horizontal separation of cracks 4.1 mm 6061-T6 aluminum alloy, stress, 25,000 psi.

After the tip of the crack had passed a certain region, little additional deformation occurred there. Sometimes, however, a small branch crack formed along the main crack in regions where some stress concentration remained. This event was slightly more common at the lower stresses, presumably because a larger number of cycles was available before the specimen failed. Cracks appearing along the flank of the main crack sometimes rejoined it, especially when the new crack could cut across a loop in the original crack. Branches were more likely to complete the bridging process at higher stresses. On rare occasions, a piece of metal that had been surrounded on all sides by cracks was undermined by their deepening and fell from the specimen, leaving a hole in the surface.

As cracks grew they exerted a pronounced influence on other cracks in their vicinity. A small crack did not continue to grow appreciably after a large crack had passed close by. Also, the rate of growth of two large cracks decreased when they came abreast of each other traveling in opposite directions. This is illustrated in Fig. 4, where the lower portion of crack *A* came opposite the upper portion of crack *B* at 400,000 cycles. The growth of the near ends of these two cracks was immediately slowed, while that of the free ends continued unchanged. Sometimes two overlapping but unjoined cracks were found which together acted as one large crack. In some cases, the progress of a crack was slowed when a new crack was formed ahead of it at such an angle as to relieve part of its stress field.

Just before a specimen failed, much deformation commonly occurred at the end of a crack, giving it a feathered appearance. This was partly the result of stress concentrating near the crack and partly the result of increased bending of the specimen because of the deep crack. Just before failure, the crack sometimes showed a slightly increased rate of growth. The length of the crack immediately before failure, however, corresponded exactly to that region of the fracture surface containing typical fatigue markings, demonstrating the accuracy with which fracture surface observation indicates fatigue history. The fatigue portion of the fracture surface was divided roughly into two zones: one darkened by abrasion product and the other having the color of clean aluminum. The latter zone proved to be the metal that cracked in about the last 10,000 cycles before failure.

Rate of Crack Propagation:

Many equations have been proposed for the growth of a fatigue crack. Wilson and Burke (4), Phillips (5), and discussion by Weibull (5), all using large, plate-type specimens, suggested that there is a straight-line relationship between the crack length and number of cycles. Bennett (6), on the other hand, found a straight line resulted only when the logarithm of the crack length (from which a small constant was subtracted) was plotted against the number of cycles on a linear scale. Head's theory (3), which has some experimental support (7), deduced that the inverse square root of the length should be proportional to the number of cycles. This plots as a straight line on log-log scales.

Though the data obtained in this investigation were such that in certain ranges there was little to choose between the different methods of plotting, the bulk of the data fitted Bennett's relation best. Except at the highest stresses, the points fell well along straight lines when the logarithm of the crack length was plotted against the number of cycles on a linear scale. At the high stresses, each crack initially began to grow according to Bennett's relation, but so many cracks formed that soon their mutual interference began to change their individual rates of progression. It was found, however, that the total length of all cracks eventually contributing to failure at these stresses was approximately a linear function of the number of cycles.

If it is assumed that the Bennett-type plot is best (neglecting his small subtractive constant) the equation relating the length of the crack, l, to the number of cycles, N, is of the form

$$\log l = aN + b \ldots\ldots\ldots\ldots (1)$$

where *a* and *b* are constants. This means that at any stage in the growth of a crack, a constant number of cycles is needed for it to increase in length by a given multiple. The concept of "doubling time," the number of cycles needed for the crack to double in length, is introduced as an easily visualized quantity. During each interval of doubling time, the crack doubles in length.

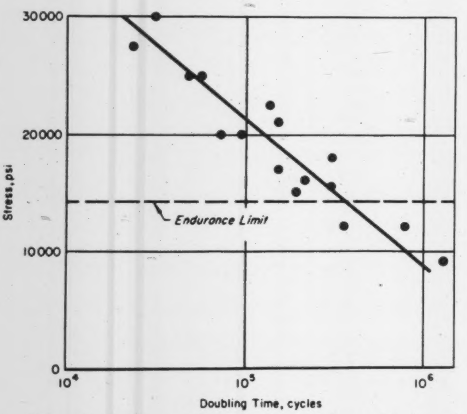

FIG. 5.—Effect of Stress Level on Rate of Growth of Fatigue Cracks in 6061-T6 Aluminum Alloy. Rate of growth is given in terms of doubling time, the number of cycles needed for a crack to double in length. Points below the endurance limit were obtained from pre-cracked specimens.

In Fig. 5, the rates of propagation for specimens at the various stress levels are plotted in terms of the doubling times. Although there is much scatter, it will be seen that the points lie generally along a straight line when the stress is plotted on a linear scale and the doubling time on a logarithmic scale. Bennett (6) has reported a direct proportionality between the two quantities in the case of a steel over a smaller stress range than that of Fig. 5. Head's theory (3) proposes a complex relation through the term $S^3(S_y - S)^{-2}$, where S and S_y are the fatigue stress and yield strength, respectively.

In general, each end of a crack measured from the origin had the same doubling time as the crack as a whole and each portion acted more or less independently. If one end of a crack became halted temporarily the other end continued growing at the same rate. When the halted end resumed growing, it again assumed its original rate (see, for example, the upper portion of crack *B* in Fig. 4).

For some reason, as yet unexplained, there was a decided tendency for both ends of a crack to stop temporarily when the crack reached a critical length of about 0.3 mm. After several millions of cycles (the time being longer at the lower stresses), the crack again continued to grow with much the same rate as it had before. The interval over which crack growth remained checked was largely unpredictable so that those specimens in which the main crack stopped the longest time had the largest spread between the first crack and failure points. The spread between the First Crack and Failure curves depends, therefore, on both the rates of crack propagation at the various stress levels and the intervals over which cracks do not grow.

The critical length for the stopping of small cracks coincides almost exactly with the finite length with which Bennett's cracks in X4130 steel seemed to start (6). This suggests that his cracks, in fact, started with lengths unresolvable by his technique and that the sensitivity of cracks of this dimension to stoppage may be a universal property of all metals.

Propagation Below Endurance Limit:

Some specimens were run at stresses above the endurance limit until a crack was formed and were then tested at stresses below the endurance limit. It was found that, in general, the cracks would grow at these low stresses at rates that were simple extrapolations of the rates above the endurance limit. Figure 5, in fact, includes several points for rates below the endurance limit. The stress level at which the crack had been pre-formed had no discernible effect on the growth rate, but when more than one crack was present only one crack grew. In no case did the crack resume growing at the new rate as soon as the stress was reduced; rather, an appreciable number of cycles was needed at the new stress before the crack began to enlarge. The length of time required varied according to the stress level: the lower the new stress, the longer the time.

There does exist a stress below which cracks will not propagate, at least within practical time limits. In this connection, the behavior of one particular specimen is interesting. A large crack 8.5 mm (0.33 in.) long was formed in this specimen at 18,000 psi. The stress was then reduced to 6000 psi. At this stress, the ends of the crack had not extended after 65 million cycles; however, a number of small branch cracks formed along the crack sides even at this very low stress. When the stress was raised to 9000 psi, more side branches formed until the crack started to grow (though at one end only) after 17 million cycles. It appears that the over-all stress must be reduced to a very low level to prevent any additional cracking in a cracked specimen, provided cycling is to be continued for a very long time.

When the stress on a cracked specimen was reduced to a level at which growth did not occur and was subsequently raised to the original level, growth resumed immediately and at the original rate. The intermediate cycling at the low stress served only as an interruption in the test.

CONCLUSION

The propagation of fatigue cracks is a complex phenomenon, especially at high stresses where there are a large number of cracks that mutually influence each other. Fatigue cracks, moreover, may give rise to entirely new cracks. These new cracks make their own contribution to the hodgepodge of overlapping stress fields. Under these conditions, no simple equation can be expected to be completely satisfactory in describing the growth of fatigue cracks. The exponential-type proposed by Bennett seems to be the best of those available. Fatigue cracks grow faster (in terms of distance traveled per cycle) as they grow larger.

The rate of crack growth is not directly proportional to the stress. Cracks at low stresses propagate much more slowly than those at high stresses. Cracks may propagate at stresses even below the endurance limit, indicating that the endurance limit phenomenon is controlled primarily by factors associated with crack initiation rather than with crack growth. Much of the scatter of fatigue testing is related to scatter in crack initiation, although apparently a random element is also involved in whether a propagating crack becomes temporarily immobilized.

The factors involved in the immobilization of a crack are not completely understood. This is especially true in the case of cracks 0.3 mm long which seem unusually prone to immobilization. Another point that is not fully explained is why cracks do not continue growing immediately when the test stress is reduced. If these factors were completely understood, it might be possible to design alloys or structures having a low susceptibility to fatigue crack propagation.

Acknowledgment:

The authors express their appreciation to F. Keller under whose direction this investigation was conducted and to B. A. Walker of the Mechanical Testing Division of the Alcoa Research Laboratories who conducted the fatigue tests.

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DISCUSSION

Mr. Frank A. McClintock.[1]—The authors are to be congratulated on their very detailed and interesting set of observations. Could they give a reference to, or describe their chemical polishing and replica techniques in more detail?

Perhaps the authors' conclusion that branches are always the result of joining up of cracks depends on the location of the crack and the type of stress field. Work to be reported on torsion in which the cracks were observed by sectioning[2] showed extensive branching but no evidence of cracks in advance of the main crack joining up with it.

Mr. John A. Bennett.[3]—I have a few questions and comments regarding the interesting results shown in this paper.

The effect shown in Fig. 2 is rather unusual, as the plastically deformed region appears to be limited to the plane of the crack. I wonder if the authors could give the direction of the crack relative to the specimen axis; it seems to me that this type of deformation could occur only if the crack were nearly parallel to a plane of maximum shear stress.

The branching and discontinuous propagation of the fatigue cracks can be more easily understood when one considers the stress field around a crack under a tensile load. Post,[4] for example, showed that the maximum normal stresses at a given distance from the root of a crack do not occur directly ahead of the crack but at angles of about 65 deg to this direction. The line of action of these maximum tensile stresses is nearly perpendicular to the crack. Therefore, except as this stress pattern is modified by crystal orientation and plastic deformation, there are always two equally probable directions in which the next portion of the crack can occur.

The observation that the crack growth stopped temporarily at a length of 0.3 mm is surprising. I would like to ask the authors if this length corresponded to the first change in direction of the crack and also if both ends of the crack resumed growing about the same time. In this connection I should point out that in the work on steel to which the authors referred, I found cracks of a finite initial length only at relatively low stresses. When the stress was more than about 10 per cent above the fatigue limit, the cracks were more numerous, and those as small as 0.1 mm could be identified by the technique used.

A Member.—Perhaps we should emphasize the effect of specimen geometry and change in stress for the practical engineers on the rate of crack growth. While we do not have a great deal of data, we have found that for cracks under 10 mm the crack growth appeared to be almost linear against the number of cycles. Once the crack length becomes appreciable, it grows at an increasing rate. The crack cessation at 0.3 mm is surprising because it comes roughly at the size we have noted with some non-progressing cracks in specimens with holes, where they stop growing somewhere between the range of about 0.3 mm and 0.16 mm from the hole. Of course, the crack probably starts slightly down in the hole, and we are only observing the surface crack.

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[1] Massachusetts Institute of Technology, Cambridge, Mass.

[2] Frank A. McClintock, "The Growth of Fatigue Cracks under Plastic Torsion," International Conference on Fatigue, Institution of Mechanical Engineers, London, Sept. 1956.

[3] Chief, Mechanical Metallurgy, National Bureau of Standards, Washington, D. C.

[4] Daniel Post, "Aircraft Glazing Materials; Photoelastic Stress Analysis for an Edge Crack in a Tensile Field," Naval Research Laboratory Memorandum Report No. 175, June 1953.

I would like to ask if the authors have observed any non-progressing cracks in their unnotched specimens. We have noted in one case a couple of very small ones in a polished unnotched 1015 steel specimen, but I suspect these are due to peculiar metallurgical phenomena.

MR. R. C. A. THURSTON[5] (*by letter*).—The authors are to be commended for their continuing research into the more fundamental aspects of fatigue in aluminum alloys and in particular for their intensive study of crack propagation as reported in the present paper. Having been associated with a somewhat similar project, the writer is aware of the countless weary hours that must be spent in accurate measurement and recording in an investigation of this nature.

Two aspects of their experimental results were of particular interest, the first being the tendency for cracks to stop propagating at a critical length of about 0.3 mm, and the second being the relationship between the stress and the logarithm of the crack doubling time. With regard to the former, the authors state that the crack continued to grow after several million cycles. In view of the *S-N* curves in Fig. 1, this fact would restrict the observations to the low stress region (below 20,000 psi), where the endurances are of a sufficiently high order. Would it be correct then to say that no "halting" cracks were observed at the higher stresses? Furthermore, have the authors any information concerning the grain size of the material?

As regards the second point, the authors found that cracks formed above the endurance limit propagated at lower stresses at rates that were simple extrapolations of the rates above the limit. Frost[6] and Phillips,[7] on the other hand, found no evidence of continuous slow crack growth at stresses below the endurance limit, and in fact no apparent growth up to within a few thousand cycles of complete failure.

Their materials were two steels and a 72,000-psi aluminum alloy (L65), water quenched and aged. In addition they reported that if the nominal stress applied to a precracked specimen was reassessed on the basis of the reduced cross-section and the unsymmetrical loading conditions, the values obtained plotted relatively smoothly on a new *S-N* curve. The endurance limit of the precracked material as determined from this curve, was about 20 per cent of the ultimate tensile strength in each case. Applying this result to the authors' principal material gives an estimated endurance limit for the cracked specimens of ± 8900 psi. One of their specimens with a crack about 0.33 in. long was tested below the normal endurance limit at a stress of 6000 psi and showed no sign of crack extension after 65×10^6 cycles. If the stress is reassessed along the lines mentioned above, a reasonable approximation would be 8300 psi and the behavior of the specimen is thus in line with what might be predicted from the work of Frost and Phillips. Subsequently the stress was raised to a nominal value of 9000 psi reassessed at 12,500 psi and the crack began propagating, presumably to failure as would be expected.

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[5] Head, Engineering Physics Section, Department of Mines and Technical Surveys, Physical Metallurgy Div., Ottawa, Ont., Canada.

[6] N. E. Frost, "Crack Formation and Stress Concentration Effects in Direct Stress Fatigue," *Engineer*, Vol. 200, pp. 464–467, 501–503 (1955).

[7] Reference (**5**) of paper. See also: E. Kilbride and C. E. Phillips, "Researches on Fatigue of Metals at Mechanical Engineering Research Laboratory," *Transactions* Inst. of Eng. and Shipbuilders in Scotland, Vol. 99, pp. 173–192 (1955–1956).

It would be interesting to know if the authors have any additional results which might help to prove or disprove the foregoing hypothesis.

MESSRS. M. S. HUNTER AND W. G. FRICKE, JR. (*authors' closure*).—Chemical polishing of the specimens used in this investigation was performed using the Alcoa R5 bright dip process. This is a patented process involving a mixed acid reagent, and licensed by the Aluminum Company of America.

Faxfilm, a former product of the Brush Development Co, was used as the replica material. A plastic tape, when moistened by the solvent provided, assumes the contour of the specimen surface. The replication is extremely good and suitable for the production of electron microscope replicas if desired.

It should be reemphasized that this investigation treated only of the spread of fatigue cracks as seen at the surface of the specimens. Nothing in the paper should be construed as indicating mechanisms of growth beneath the surface. It is therefore not surprising that McClintock's sectioning experiments disclosed no evidence of cracks forming in advance of a deepening crack.

In response to Mr. Bennett's question concerning the direction of the stress relative to the crack shown in Fig. 2 of the paper, it will be noted that there are a series of parallel diagonal lines in the photograph. These are minor circumferential scratches. The angle in the surface plane between the direction of the crack and the nominal direction of stress is 40 deg. A faint line in this figure which lies at an angle of 27 deg to the crack (67 deg to the stress) has the appearance of a plastically deformed region on the verge of becoming a branch crack. Such extreme cases of plastic deformation were found only in a few specimens of X7375 aluminum alloy.

The existence of hesitating or nonpropagating cracks is an interesting phenomenon. Perhaps several types should be differentiated. One is the type described by Frost,[6] Phillips,[7] or one of the present discussers where a crack originates in a stress raiser such as a notch or hole and stops growing when its tip passes out of the "field" of the stress raiser. This type is not described in the present paper. A second case of interrupted growth occurs when the stress on a propagating crack is suddenly reduced. It has been thus far assumed that the failure of the preexisting cracks in Phillips' specimens to grow until shortly before failure was closely analogous to our observation of the long periods of time elapsing before the cracks in precracked specimens resumed growing at the rate typical of the new stress.

The third case of interrupted propagation is probably the most interesting of all. This is the case where small cracks halt temporarily in their growth upon reaching a rather critical size range. Little can be advanced as yet to account for the phenomenon. Because the crack has changed direction several times before it reaches immobilization size, Mr. Bennett's suggestion is inapplicable. Likewise, Mr. Thurston's suggestion of a correspondence between grain size and critical length seems unlikely because the immobilized crack extends across roughly five grains. (In the material used, the average grain dimension was 0.091 mm for all directions but 0.067 mm in the direction perpendicular to the stress.)

One possible explanation which we have as yet been unable to investigate is that the initial, subcritical cracks are aided in their formation by the vibrations occasioned by minor inescapable misalignments of the specimen in the testing machine. Once cracks have grown to critical size, the inherent surges are smoothed out by the presence of the cracks themselves so that continued growth must depend on the normal fatigue process.

The length of time during which the cracks become immobilized varies from 10^5 cycles at 20,000 psi to 10^7 cycles at stressses slightly above the endurance limit. At higher stresses, the hesitation times are so short that they become difficult to measure and, moreover, they consume only a minor portion of the fatigue life. This again is in line with Mr. Bennett's observation that cracks with finite initial length are found only at low stresses. Once the immobilized crack resumes growing, it starts propagating from both ends almost simultaneously.

The question as to how the stress distribution within a specimen changes as fatigue cracks are initiated and grow is difficult to answer. One does not know, for example, if a crack will support a compressive stress across it or, even, if the ruptured material loses all of its ability to support a tensile load. The situation is complicated, moreover, by differences in testing loads. At low test stresses, one dominant crack is formed which, as it grows, causes the neutral bending plane to shift away from the original specimen axis and probably to oscillate as the specimen rotates. At high stresses, on the other hand, cracks quickly form at many points around the circumference so that the neutral plane is little disturbed and only the effective cross-sectional area changes.

In light of these complications, it is rather surprising that crack geometry did not affect our test results more than it did. It was, in fact, found that cracks did not change their initial rate of propagation, which was ordained when their size could hardly have affected the loading distribution, until they grew to such size as to interact with their neighbors. The only effect noted was that in specimens containing only one dominant crack a sudden acceleration of growth was sometimes noted when the crack reached huge proportions, say occupying half of the circumference.

At the present time, we have no additional information concerning Mr. Thurston's calculations as to the location of the endurance limit for the precracked material.

THE NOTCHED FATIGUE PROPERTIES OF SOME TITANIUM ALLOYS*

BY A. W. DEMMLER, JR.,[1] M. J. SINNOTT,[2] AND L. THOMASSEN[2]

SYNOPSIS

A study has been made of the notched rotating-beam fatigue life characteristics of three commercial titanium alloys: Ti-75A, RC-130B, and RC-A-110AT, and two experimental alloys: 6 per cent aluminum (an all-alpha alloy) and 30 per cent molybdenum (an all-beta alloy). The influence of various notch shapes and of different means of preparing these notches has been evaluated. Machined notches decrease the fatigue properties less than would be predicted by K_T, the theoretical stress concentration factor. Rolling the notch after machining appears to remove the strength reduction effect of the notches. Ground notches produce reductions in strength equal to or greater than those which would be predicted by K_T.

The present investigation extends the work previously reported on the fatigue properties of titanium alloys in the unnotched condition to the properties of these same alloys in the notched condition (1).[3] Various types of notches of different severity were tested with the additional parameter of the method of notch preparation also being investigated. A replicate testing procedure was followed in this work in order to obtain a more reliable estimate of the fatigue life characteristics.

TEST MATERIALS AND TEST SPECIMEN PREPARATION

Table I lists the analyses of the various alloys used in this investigation. With the exception of the heats labeled all-alpha and all-beta the materials used were commercial heats of titanium alloys.

The test specimens were made from $\frac{5}{8}$ or $\frac{1}{2}$ in. round stock supplied in the hot-rolled or swaged and annealed condition. All materials at some time prior to testing were given an additional anneal at 1450 F in argon to remove any residual stresses that may have been present in the as-received stock. Tests made previously showed that there were no significant variations between or within bars of a given heat, so variations from this source may be neglected (1).

Table II lists the mechanical properties of the various alloys that were tested. Microstructural examination showed no significant differences between different heats of the same alloy.

Test Specimen Preparation:

All the fatigue testing was carried out on rotating beam R. R. Moore type machines. The different types of test specimens that were used are shown in Figs. 1 to 7. The specimen as shown in Fig. 1 was prepared by machining the test specimens to within 0.005 in. of the final dimensions, annealing for $\frac{1}{2}$ hr in argon at 1450 F, then hand finishing with metallographic papers through No. 000 grit to the final dimensions. This surface finish was taken as the unnotched surface and the notched fatigue life values were compared to this as a standard. The specimens shown in Figs. 2 to 6 are V-, square-, and radius-notch specimens. The notch shapes are the same in this group of specimens, but the minimum diameter of the test specimen was varied. This was found to be necessary in order to be able to apply sufficient stress to the specimens in order to produce fracture at the stress levels used.

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[1] Research Engineer, Physical Metallurgy Division, Alcoa Research Labs., Aluminum Company of America, New Kensington, Pa.

[2] Professor of Metallurgical Engineering, Univ. of Michigan, Ann Arbor, Mich.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1060.

In some specimens, Figs. 5 and 6, the diameter of the main cylindrical section of the specimen was reduced to decrease the fretting corrosion at the mouth of the female taper as well as to prevent certain spurious results in the form of cracking near this point.

TABLE I.—CHEMICAL ANALYSES OF ALLOYS, PER CENT BY WEIGHT.

Material	Heat Number	Carbon	Nitrogen	Iron	Tungsten	Hydrogen	Manganese	Aluminum
Ti-75A..........	M-270.......	0.04 to 0.07	0.10	0.20	0.01	0.02 to 0.05	(Oxygen = 0.20)	... ...
RC-130B........	B-3413.......	0.1	0.08	...	...	...	3.9	3.9
RC-130B........	B-5331.......	0.1	0.04	...	...	...	4.4	3.9
RC-A-110AT....	R-30314......	0.1	0.05	...	Tin 2.58	...	...	4.13
All-Alpha.......	.....	0.06	0.035	...	...	...	...	5.78
All-Beta.........	6189........	0.016	0.069	...	...	Molybdenum	29.88	...
	6188........	0.011	0.065	...	...	Molybdenum	30.17	...

TABLE II.—MECHANICAL PROPERTIES OF ALLOYS.[a]

Material	Heat Number	Proportional Limit, psi[b]	Tensile Strength, psi	Elongation in 2 in., per cent	Reduction of Area, per cent
Ti-75A..............	M-270..............	47 900	84 300	27.3	42.0
RC-130B............	B-3413.............	119 000	160 000	18.0	47.0
RC-130B............	B-5331.............	138 000	148 000	18.7	46.0
RC-A-110AT.........	R-30314............	124 000	138 000	16.0	40.0
All-Alpha............	.....	81 900	131 500	10.1	33.5
All-Beta.............	6188...............	112 000	128 750	7.8	33.2
	6189...............	110 000	120 000[c]	2.0[c]	1.0[c]
RC-130B[d]...........	B-3413[d]...........	158 000[d]	186 000[d]	2.8[d]	1.0[d]

[a] Duplicate tests. Test specimen diameter, 0.438 in.
[b] SR-4 strain gages used.
[c] Fracture in gage marks. Single test.
[d] Water quenched from 1550 F, tempered 2 hr at 700 F (fracture in the gage marks).

The notched portions of the machined V-, square-, or radius-notch specimens labeled VM, SM, or OM, were machined to within 0.010 in. of their final size, annealed in argon at 1450 F for $\frac{1}{2}$ hr, then finish machined with a pre-contoured tool. The notches prepared by diamond grinding (DG) had been, prior to annealing, machined to 0.007 in. greater than the final diameter. These specimens were forwarded to a commercial diamond grinding shop for finishing. Light feeds were used, and an excellent finish and dimensions were obtained in this process. The diamond grinding was thought to be necessary since early attempts at grinding using the conventional aloxite or silicon carbide wheels were unsuccessful. Later a technique was developed for grinding with an aloxite type A80V2BT wheel. For this grinding, a 6-in. diameter wheel rotating at 5200 rpm counter-clockwise was fed to the work in steps of 0.001 in. The work rotated clockwise at a speed of 1100 rpm. Afton No. 8 oil of the Tidewater Oil Co. was used as a coolant. This grinding operation (CG) produced a good surface and root radius on the notches with very slight wheel wear.

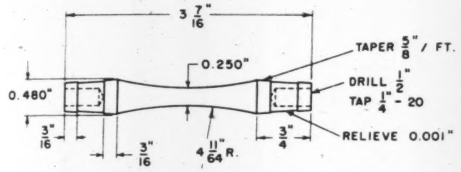

Fig. 1.—Unnotched Fatigue Test Specimen.

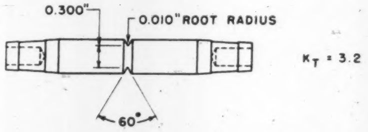

Fig. 2.—60 deg V-Notch Fatigue Test Specimen.

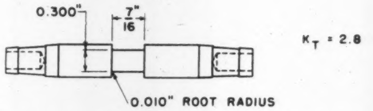

Fig. 3.—Square-Notch Fatigue Test Specimen.

Most of the notch specimens were checked against a prepared template with a micro-projector in order to insure that the root radii of the notches were maintained. In no case did the root radius appear to be in error by as much as 0.001 in.

Since the previous work had shown that shot-peening the surfaces of the unnotched specimens improved the fatigue properties, it was believed that a surface rolling of the V-notched specimens would improve the notch fatigue strength of these same alloys. For this purpose, annealed machined notch specimens, 0.005 in. oversize in the notch, were rolled by adapting a pipe cutting tool in such a manner that the cutting tool would roll the notch to the finished size and shape. Notches prepared in this fashion are reported as rolled notches (VR).

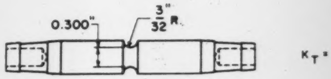

Fig. 4.—Radius-Notch Fatigue Test Specimen.

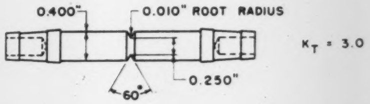

Fig. 5.—Reduced Diameter V-Notch Fatigue Test Specimen.

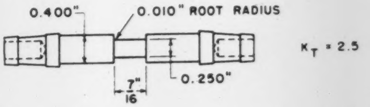

Fig. 6.—Reduced Diameter Square-Notch Fatigue Test Specimen.

Since the surface rolling improved the fatigue life characteristics of the alloys, a still more severe rolling operation was used to produce a V-notch on the RC-130B alloy. The specimens, shown in Fig. 7, were machined as cylinders with no premachining of the notch. The notch was then rolled directly to size using the contoured pipe-cutting tool. In both this

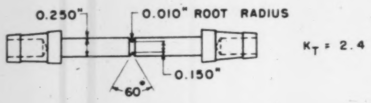

FIG. 7.—Heavy-Rolled V-Notch Fatigue Test Specimen.

operation and in the lighter rolling operations, no difficulty was encountered in producing these notches and in maintaining the root radius. Surfaces were good with some slight surface checking occurring, particularly in the alloys.

TABLE III.—SUMMARY OF FATIGUE TESTS.

Material	Test Surface[a]	Speed of Testing, rpm	Number of Specimens	Number of Stress Levels	Specimen Type
Ti-75A M-270	HF	1 800	24	4	No. 1
	HF	10 000	36	6	No. 1
	VM	6 000	15	5	No. 2
	SM	6 000	15	5	No. 3
	OM	6 000	18	7	No. 4
	VR	6 000	12	5	No. 5
	VDG	6 000	12	4	No. 2
RC-130B B-3413	HF	6 000	18	4	No. 1
	VM	6 000	15	5	No. 2
	SM	6 000	14	5	No. 3
	OM	6 000	14	5	No. 4
	Q and T, VM	6 000	15	5	No. 2
	Q and T, SM	6 000	13	5	No. 3
	Q and T, OM	6 000	5	3	No. 4
B-5331	HF	6 000	12	6	No. 1
	Q and T, HF	6 000	9	3	No. 1
	VR	6 000	15	6	No. 5
	VHR	6 000	13	6	No. 7
	VDG	6 000	14	6	No. 2
	VCG	6 000	14	5	No. 5
	SCG	6 000	13	5	No. 6
RC-A-110AT R-30314	HF	6 000	13	5	No. 1
	VM	6 000	15	7	No. 2
	SM	6 000	14	4	No. 3
	VR	6 000	14	6	No. 5
	VDG	6 000	14	5	No. 2
	SCG	6 000	13	4	No. 6
Alpha	HF	6 000	16	6	No. 1
	VM	6 000	15	5	No. 5
	SM	6 000	15	5	No. 6
	VR	6 000	13	6	No. 5
	VCG	6 000	15	5	No. 5
Beta 6188 and 6189	HF	6 000	11	6	No. 1
	VM	6 000	12	6	No. 5
	SM	6 000	11	4	No. 6
	VR	6 000	11	5	No. 5

[a] HF = Hand finished.
VM = Machined V.
SM = Machined square.
OM = Machined radius.
VR = Rolled V.
VHR = Heavily rolled V.
Q and T = Quenched and tempered.
VDG = Diamond ground V.
SDG = Diamond ground square.
VCG = Commercially ground V.
SCG = Commercially ground square.

The notch types investigated on each alloy, the number of stress levels investigated, and the numbers of specimens used to determine the S-N curve for each type of notch are listed in Table III.

Notch Characteristics:

There are three common ways of describing the notch fatigue characteristics of a material (2). The first is by the use of the theoretical stress-concentration factor, K_T. This describes for an ideal, homogeneous, isotropic, and elastic material the ratio of the maximum local stress to the nominal stress. The values are obtained from theory and photoelastic studies. Another method of describing the effect of a notch on the fatigue preoperties is to use what is termed the fatigue reduction factor, K_f. This is numerically equal to the ratio of the fatigue strength of an unnotched specimen at N cycles of stress to the fatigue strength of a notched specimen at N cycles of stress. It will be noted that K_f can vary with fatigue life while K_T is a constant depending only on the shape of the specimen. It has been generally noted that $K_f < K_T$. A third method of describing notch sensitivity is the use of a q index. q is defined as equal to $(K_f - 1)/(K_T - 1)$. With this criterion, a notch not resulting in any strength reduction will make $q = 0$. Similarly, if $K_f \geqq K_T$, then $q \geqq 1.0$. The K_T values for the various specimens are shown in the sketches of the specimens and have been taken from the literature (3, 4).

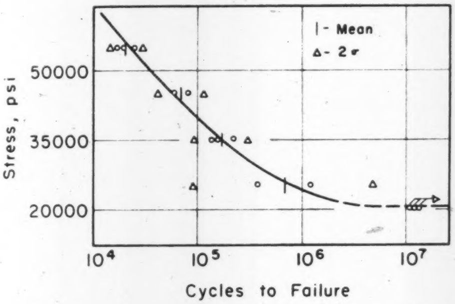

FIG. 8.—Fatigue Data, Ti-75A, Heat M-270, Diamond Ground V-Notch (VDG).

Fatigue Testing:

Identical tests made on each of five R. R. Moore fatigue machines showed that there was no significant difference between the performance of the various units. The majority of the specimens were run at a speed of 6000 rpm. Previous work had shown that the effect of speed of testing in the range from 1800 to 10,000 rpm had only a slight effect on the fatigue life characteristics.

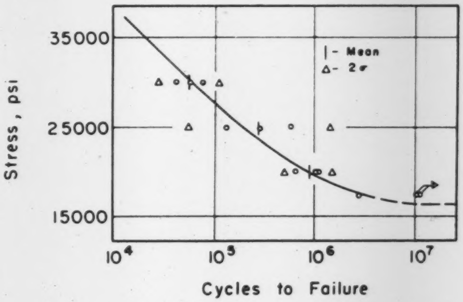

FIG. 9.—Fatigue Data, RC-130B, Heat B-5331, Square Notch, Commercially Ground (CG).

EXPERIMENTAL RESULTS

In general, three replicate tests were made at each of five stress levels for each notch type studied in a particular alloy. As in the previous paper, an attempt was made to space these stress levels in such a manner as to obtain fatigue life values between 10^4 and 10^7 cycles. In some of the experimental heats only duplicate tests were made because of the lack of sufficient stock. Table III lists the number of test specimens and stress levels investigated for each notch and alloy type.

Figures 8 and 9 are typical stress-

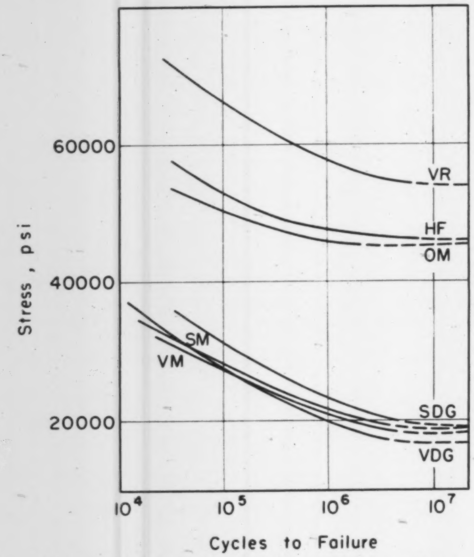

FIG. 10.—Summary Graphs of Fatigue Data, Ti-75A, Heat M-270.

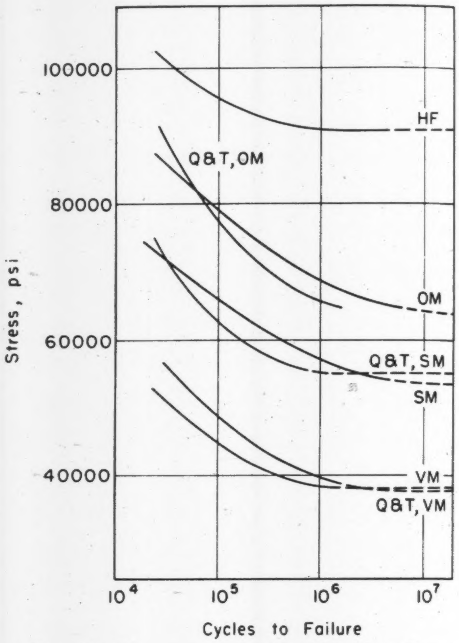

FIG. 11.—Summary Graphs of Fatigue Data, RC-130B Alloy, Heat B-3413.

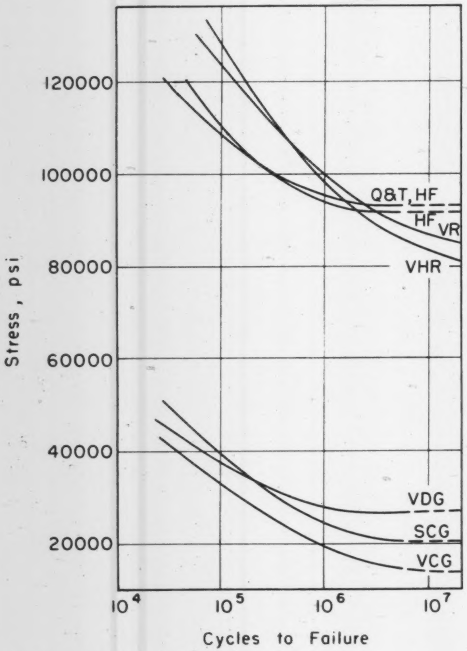

FIG. 12.—Summary Graphs of Fatigue Data of RC-130B Alloy, Heat B-5331.

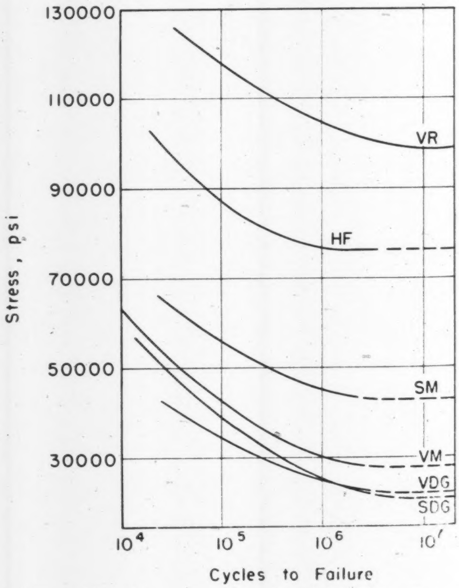

FIG. 13.—Summary Graphs of Fatigue Data, RC-A-110AT Alloy.

cycles of stress data obtained in the testing program. The data at the higher stress levels appear to be more closely grouped about the mean than at lower levels. In determining the mean values of a group, a log-normal distribution of cycles to failure was assumed in view of the other findings on the statistical nature of fatigue (5). The standard deviation from the mean is that of the universe and is computed by methods outlined in standard texts on statistics (6).

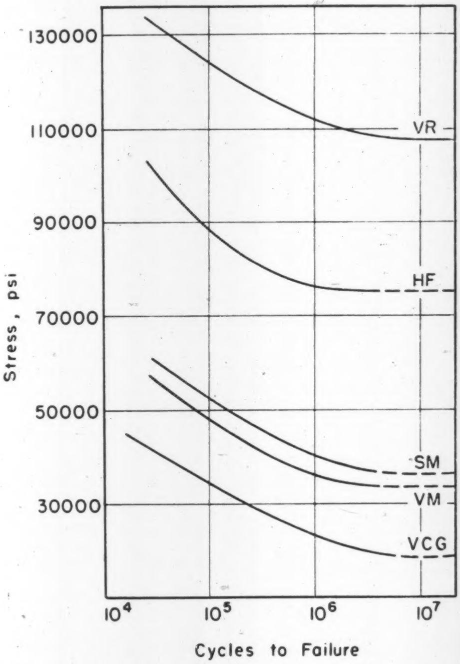

FIG. 14.—Summary Graphs of Fatigue Data for All-Alpha Alloy.

Figures 10 to 15 summarize the data of this research and show curves of best fit through the mean values as determined from the data presented in curves similar to those shown in Figs. 8 and 9. As such, these curves represent the best approximation to the S-N curve which would result from testing a universe of specimens rather than a sample of thirty or less which has been tested. While the curves represent the mean fatigue life, the curve as it approaches 10^7 cycles of stress is an estimate of the endurance limit of the alloy. This latter value is extrapolated slightly in a number of cases, although it has frequently been determined by three run-outs. With tests of the type outlined, the value of the endurance limit could not be satisfactorily determined statistically. It is expected that any small errors made in the extrapolation of the strengths at 10^7 cycles have been made on the low or safe side for the material studied. This procedure has been continued in order to maintain the continuity with the previous paper (1).

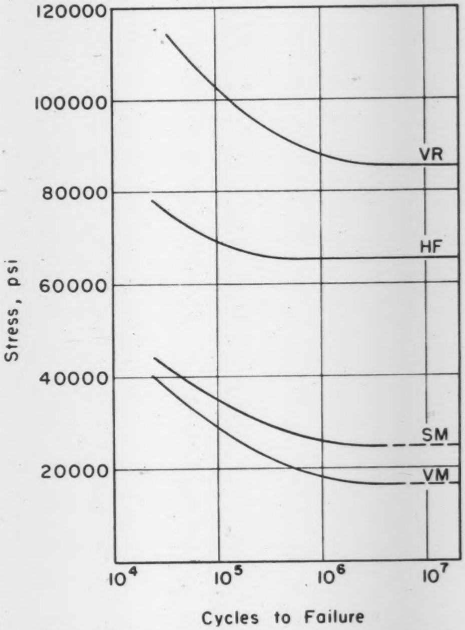

FIG. 15.—Summary Graphs of Fatigue Data, All-Beta Alloy.

DISCUSSION

Figure 10 shows the S-N curves for the unnotched and notched specimens of

Ti-75A. The absence of any strength reduction effect caused by surface rolling the machined V-notch is quite noticeable. The machined radius notch specimens have properties quite similar to the unnotched specimens. The machined V- and square-notch specimens had quite similar fatigue lives when compared with the diamond-ground notches of the same type, although the actual data for the diamond-ground specimens showed less variation about the mean value at a given stress level.

TABLE IV.—FATIGUE STRENGTH REDUCTION FACTORS AND NOTCH SENSITIVITY INDICES.

Alloy	Notch Type	K_T	K_f		q	
			10^5 cycles	10^7 cycles	10^5 cycles	10^7 cycles
Ti-75A	VM	3.2	1.95	2.41	0.43	0.64
	VR	3.0	0.79	0.81	−0.10	−0.09
	VDG	3.2	1.91	2.62	0.41	0.74
	SM	2.8	1.88	2.36	0.49	0.76
	SDG	2.8	1.68	2.28	0.38	0.71
	OM	1.4	1.05	0.96	0.12	−0.10
RC-130B	VM	3.2	2.14	2.38	0.52	0.63
	VM (Q and T)	3.2	1.97	2.40	0.44	0.64
	VDG	3.2	2.99	3.46	0.91	1.12
	VR	3.0	0.89	1.05	−0.06	0.25
	VHR	2.4	0.86	1.10	−0.10	0.07
	VCG	3.0	3.41	6.86	1.20	2.93
	SM	2.8	1.45	1.70	0.25	0.39
	SM (Q and T)	2.8	1.53	1.66	0.29	0.37
	SCG	2.5	2.81	4.56	1.21	2.37
	OM	1.4	1.21	1.42	0.52	1.05
	OM (Q and T)	1.4	1.23	...	0.57	...
RC-A-110AT	VM	3.2	2.05	2.74	0.48	0.79
	VR	3.0	0.74	0.77	−0.13	−0.11
	VDG	3.2	2.51	3.47	0.69	1.12
	SM	2.8	1.55	1.78	0.31	0.43
	SCG	2.5	2.21	3.68	0.81	1.79
Alpha	VM	3.0	1.86	2.25	0.43	0.62
	VR	3.0	0.71	0.70	−0.14	−0.15
	VCG	3.0	2.62	4.10	0.81	1.55
	SM	2.5	1.71	2.10	0.47	0.73
Beta	VM	3.0	2.37	3.97	0.68	1.48
	VR	3.0	0.67	0.76	−0.16	−0.12
	SM	2.5	1.95	2.62	0.63	1.08

The summary curves of Fig. 11 are for machined notch specimens of RC-130B. Some of this stock was heat treated at 1550 F, water quenched, then tempered at 700 F for 1 hr prior to machining. The purpose of this treatment was to change the structure from a 75–25 per cent alpha-beta structure to a 50–50 per cent alpha-beta structure. From the data it will be noted that this treatment did not change to any marked degree the fatigue life characteristics as compared to the annealed material, although such a treatment markedly improves the tensile properties.

Figure 12 shows the S-N data for the rolled and ground notch specimens of RC-130B. It will be noted that the rolling is more effective at the higher stress levels of the fatigue life curves and that a light surface rolling is as effective as the very heavy rolling operation. This would seem to indicate that the surface stresses are more important in removing or minimizing strength reduction than the subsurface stresses. The ground notch specimens all show a marked notch sensitivity.

The data of Fig. 13 for the RC-A 110AT alloy, Fig. 14 for the all-alpha alloy, and Fig. 15 for the all-beta alloy show the same trends as those developed in the Ti-75A and RC-130B alloys.

The values of K_T as taken from the literature and K_f and q as computed from the experimental data of Figs. 10 to 15 are listed in Table IV.

The values of K_f, as might be expected, are a function of N, the number of cycles of stress. In every case the value of K_f is greater, the greater is the value of N. In only a few cases, generally the ground notches and the machined notches on the all-beta alloy, do the values of K_f exceed K_T.

While not all notch types were investigated on each alloy, the data are sufficient to permit certain general statements. The surface rolling and heavy rolling of the notches on the various alloys appears to remove practically all strength reduction. It is only in the RC-130B that some trace of strength reduction remains and this is very slight. The rolling operations have evidently cold worked the root area of the notch to such a degree that in practically every case the notched specimens have superior fatigue properties to the unnotched specimens. The machined notches showed values of K_f that approached K_T at the longer cycles to failure periods. Here again, the properties are a reflection of the state of the metal in the root of the notch since machining produces residual compressive stresses which would tend to strengthen the root areas. Grinding, on the other hand, usually produces residual tensile stresses although the sense and magnitude of these stresses has been found to be a variable, depending almost entirely upon the grinding methods used. If tensile stresses are present then one generally would expect K_f values to be greater than K_T. Such seems to be the case in the alloys tested since, with the exception of the Ti-75A alloy, the K_f values are greater than the K_T values. In general, diamond grinding appears to be less severe than the aloxite grinding.

CONCLUSIONS

Machined notches decrease the fatigue life properties of titanium alloys but not to the extent predicted by K_T, the theoretical stress concentration factor.

A rolled notch appears to remove any strength reduction effect from the alloys tested. A light rolling operation is as effective as a heavy rolling operation.

Ground notches produce reductions in fatigue strength which are greater than those predicted by K_T probably because of residual stresses or surface discontinuities.

Acknowledgment:

The authors acknowledge the support of the major portion of this investigation by the Materials Laboratory of Wright Air Development Center under Contract AF 33 (616)-26.

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DISCUSSION

Mr. G. M. Sinclair.[1]—As mentioned in the paper, work of a somewhat similar nature was carried out at the University of Illinois as a part of a study on the effect of surface finish on fatigue strength of titanium alloys.[2] The measuring techniques and conclusions differed somewhat from those reported in the present paper and may, therefore, be of interest. Since the primary interest resided in fatigue limit rather than finite fatigue life, the staircase statistical method in testing was employed, defining as a "run out," specimens which survived twenty million cycles or more.

Briefly, through the use of microhardness measuring techniques, it was found that the various finishing methods studied such as surface cold rolling, mechanically polishing with emery, grinding, etc. introduced varying degrees and varying depths of cold work in the surface layers of the metal. Within the microhardness range of 300 to 500 Knoop[3] the fatigue limit of groups of smooth bar specimens having different surface finishes was found to be almost linearly related to the hardness of the surface layers produced by the finishing process while the influence of surface roughness and residual stress appeared to be relatively small.

In determining the effect of a notch on the fatigue behavior of these materials, it appeared that comparisons between notched bar and smooth bar results would, of necessity, have to be made for specimens having the same type of surface preparation (that is, ground smooth bar *versus* ground notch bar, etc.). When the fatigue strength reduction factors K_f were calculated on this basis for the various types of notches, the values were found to be equal to or slightly less than the theoretical stress concentration factors K_T determined from Neuber's relation.

Mr. A. W. Demmler, Jr. (*author*).—Had the requisite time and machines been available, it would have been desirable to run to a somewhat larger number of cycles than 10^7. It would also have been interesting, had there been sufficient specimens, to have used a staircase analysis for determination of the fatigue limit. However, while the object of the University of Illinois investigation was the determination of the fatigue limit, that of the University of Michigan study was the finite life region of the curve.

The hand-finished, unnotched specimens were chosen as a base line for calculations of notch effects due to the difficulty of obtaining comparable notched and unnotched surfaces for certain of the preparations. For example, it would have been extremely difficult to obtain an unnotched, circumferentially rolled specimen for comparison purposes. While it might have been desirable to have this type of data, even though a greatly increased number of tests would have been required, it is hoped that the form in which the data of this paper have been presented will prove to be of use.

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[1] Associate Professor of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

[2] G. M. Sinclair, H. T. Corten, and T. J. Dolan, "Effect of Surface Finish on the Fatigue Strength of RC 130B and Ti 140A Titanium Alloys," *Paper No. 55-A-197*, Am. Soc. Mechanical Engrs., Nov. 1955.

[3] See Tukon Tester, *Metals Handbook*, Am. Soc. for Metals, p. 96 (1948).

STRENGTHS AND FAILURE CHARACTERISTICS OF AMS 5765A (S-816) ALLOY IN DIRECT TENSILE FATIGUE AT ELEVATED TEMPERATURES*

By Charles A. Hoffman[1]

Synopsis

An investigation was conducted to evaluate the tensile fatigue behavior of wrought AMS 5765A (S-816) alloy. Temperatures of 1200, 1500, and 1600 F were investigated. At each temperature, three mean stress levels were investigated to give lives of approximately 10, 100, and 1000 hr at zero cyclic stress. At each condition of temperature and mean stress, direct tension cyclic stresses were superimposed upon the mean stress up to a maximum of approximately 98 per cent of the mean stress.

Because the stress conditions in these tests are similar to those in gas turbine blades in service, a study of the appearance of the failure area of specimens (fracture surfaces, ductility, etc.) under the various test conditions was conducted as an aid in the interpretation of service failures. In addition, micrographic studies were made to provide some insight into the relation of the fracture path to crystallographic features of this alloy.

The stresses in a turbo-jet turbine blade include centrifugal stress and superimposed vibratory and thermal stresses. The lives of turbine blades may be considerably less than might be expected on the basis of stress-rupture alone because of the presence of the vibratory stress.

In the analysis of service failures, the appearance of the fracture is usually used to indicate the cause of fracture. Vibratory stress is expected to cause the fracture to exhibit an appearance characteristic of fatigue and thereby suggest that fatigue has been a factor in causing blade failure. The appearance of a blade failure *per se*, however, is not necessarily conclusive evidence of the effect of vibratory stress on observed blade life. As an example, the data of Ferguson (**1**)[2] indicated that for the condition of mean stress and temperature investigated (22,000 psi and 1500 F respectively—these conditions approximate those existing at the failure zone of blades in a current turbo-jet engine), superimposed vibratory stress (to ±15,000 psi) caused lowered life, and given amounts caused either fatigue-type failure in the case of some alloys or caused stress-rupture appearing failures in the case of other alloys. For a given alloy, failure appearance was generally a function of amount of vibratory stress. Hence, failure appearance and time to failure under vibratory stress are probably functions of alloy and of stress and temperature.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] Lewis Flight Propulsion Laboratory, National Advisory Committee for Aeronautics, Cleveland, Ohio.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1079.

An investigation was conducted at the NACA Lewis laboratory to evaluate the elevated temperature fatigue behavior of a widely used wrought blade alloy, AMS 5765A (S-816), in tensile fatigue (vibratory tensile stress superimposed on a mean tensile stress). Because blade failure appearance *per se* might not indicate the effect of vibratory stress on engine blade failure time, macroscopic observations were made of the failures in relation to mean stress, temperature, vibratory stress, and time to failure to aid in the interpretation of service failures. In addition, metallographic studies were made to provide some insight into the relation of fracture path to crystallographic features of the alloy at elevated temperature. The effects of cyclic vibratory stress upon reduction in area and hardness at failure were investigated.

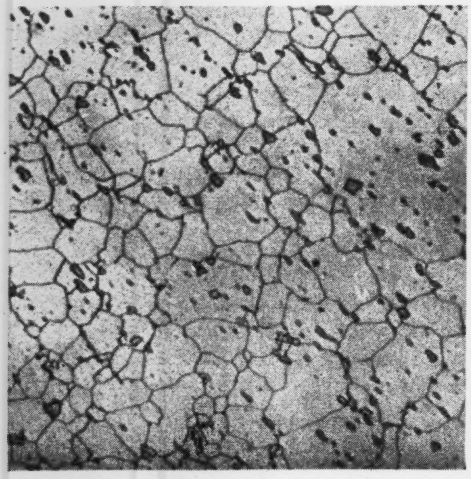

FIG. 1.—Wrought S-816 Structure After Heat Treatment, Prior to Testing. Electrolytically etched with 5 per cent solution of H_2SO_4 in 90 to 95 per cent boric acid (× 250).

MATERIAL AND SPECIMEN

The material used was wrought S-816 of the following composition, in per cent:

Carbon	0.40
Manganese	1.45
Phosphorus	0.005
Sulfur	0.008
Silicon	0.63
Nickel	19.80
Chromium	19.93
Tungsten	3.81
Cobalt	42.02
Columbium	4.07
Iron	3.35
Tantalum	0.27
Copper	0.09
Molybdenum	3.90

The material was solution treated for 1 hr at 2150 F and water quenched, then aged for 16 hr at 1400 F and air cooled. The hardness after heat treatment was 25 to 26 Rockwell C (63 to 63.5 Rockwell A). The structure after heat treatment is illustrated in Fig. 1. The grain size was in the range of ASTM No. 4–5. The surface finish on the specimen, Fig. 2, test section was 5 to 10 microinches, both longitudinally and circumferentially. The final polishing was in the longitudinal direction.

APPARATUS AND PROCEDURE

Fatigue Machines:

Commercial fatigue machines (Fig. 3) were used. The load was applied to the specimen in direct tension by a load lever arm pivoted at one end in such a manner that essentially only vertical motion was transmitted to the specimen. The load lever arm was attached at its other end to an adjustable cam. A hydraulic piston and the adjustable cam were used to adjust the maximum and minimum stresses in the specimen. The maximum and minimum stresses were determined by the deflection of the calibrated load lever arm. A deflection bar, which is shown in the storage position in Fig. 3, was placed on the load lever arm to measure deflections. Changes in the maximum and minimum deflections of the load lever arm during operation caused contacts, positioned above and below the lever arm, to be struck, which in turn actuated an electrohydraulic system to restore the desired loads. The duration of test for the specimens was indicated by electric time meters, which were automatically shut off upon failure of the specimens.

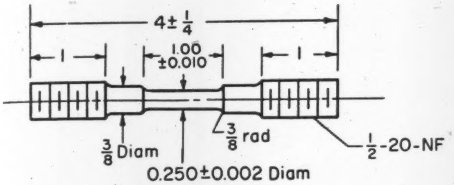

FIG. 2.—Fatigue Specimen.

Temperature Control:

The specimens were heated by resistance electric furnaces. Two couples were attached, one near either extremity of the specimen test sections. The upper (control) couple was maintained within ±5 F of the desired temperature; the lower couple was used for checks of temperature uniformity.

Evaluation Procedure:

Three temperatures—1200, 1500, and 1600 F—were used in this study. Stress-rupture data were first obtained, and from these data, mean stresses were selected to yield failure times at each temperature of approximately 10, 100, and 1000 hr with zero cyclic stress. Cyclic tensile stresses up to a maximum of approximately 98 per cent of the mean stresses were superimposed upon these mean stresses, usually in increments of ±2500 psi. The cyclic stresses were applied at 2000 cpm. The criterion of specimen failure was complete fracture.

The specimens were brought to temperature in 1 to 1½ hr and were allowed to soak for about 2 hr prior to initiation of test. About ¼ hr was required to adjust the load on the specimens. Checks were made to insure that the load settings and temperatures were properly maintained.

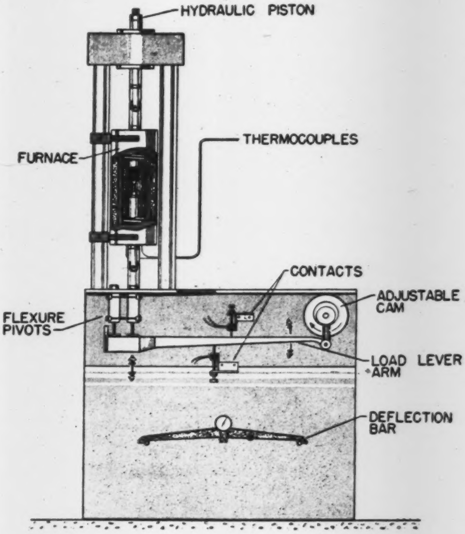

FIG. 3.—Direct Tensile Stress Fatigue Machine.

Metallographic Studies:

The fractured specimens were examined macroscopically at magnifications up to 20× and were classified according to type of failure as follows:

1. Stress-rupture.
2. Fatigue.
3. Stress-rupture plus fatigue.

Specimens that failed with zero vibratory stress were, by definition, of the stress-rupture type. Specimens run in combined stress and having failures resembling those obtained in static stress-rupture tests at corresponding mean stresses and temperatures were classified as stress-rupture failures. The macrocharacteristics of stress-rupture failure specimens are considered subsequently. Specimens were classified as fatigue failures when a large smooth facet, approximately 90 deg to the applied load, appeared in the fracture; adjacent to the facet was a zone resembling the fracture surface obtained in a tension test. Specimens having a fatigue facet and an adjacent area resembling that obtained in a stress-rupture test were identified as stress-rupture plus fatigue. In some cases it was difficult to determine clearly whether the failure adjacent to the fatigue facet was of the stress-rupture or abrupt tensile type; classification into the third category might be subject to individual interpretation.

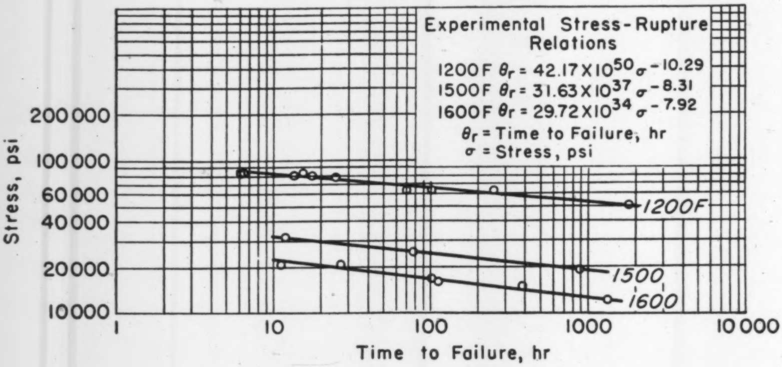

FIG. 4.—Stress *versus* Time to Failure.

Microspecimens were mounted to show a longitudinal section through the stress-rupture, fatigue, and abrupt tensile failure zones where each was present. The microspecimens were electrolytically etched with a 5 per cent solution of H_2SO_4 in 90 to 95 per cent boric acid. The fractured specimens were examined microscopically and classified according to fracture path as follows:

(1) Intergranular fracture, defined as one occurring along the boundary between grains.

(2) Transgranular-shear fracture, defined as one which occurred along crystallographic planes of approximate maximum shear stress, that is, about 45 deg to the applied load.

(3) Transgranular-cleavage fracture, defined as a fracture that occurred along crystallographic planes of approximate maximum normal stress, that is, about 90 deg to the applied load. The direction of these planes of fracture varied somewhat with grains because of differing crystal orientations.

(4) Transgranular-noncrystallographic fracture, defined as a fracture which proceeded along a path that was comparatively independent of crystallographic orientations. This path was essentially at 90 deg to the applied load.

The diameters of the failed specimens were determined at the failure zone. These data were used to compute the per cent reduction in area at failure. The hardness measurements were taken in a transverse cross-section approximately $\frac{1}{8}$ in. below the fracture surfaces and on the same half of the specimens from which the microspecimens were taken.

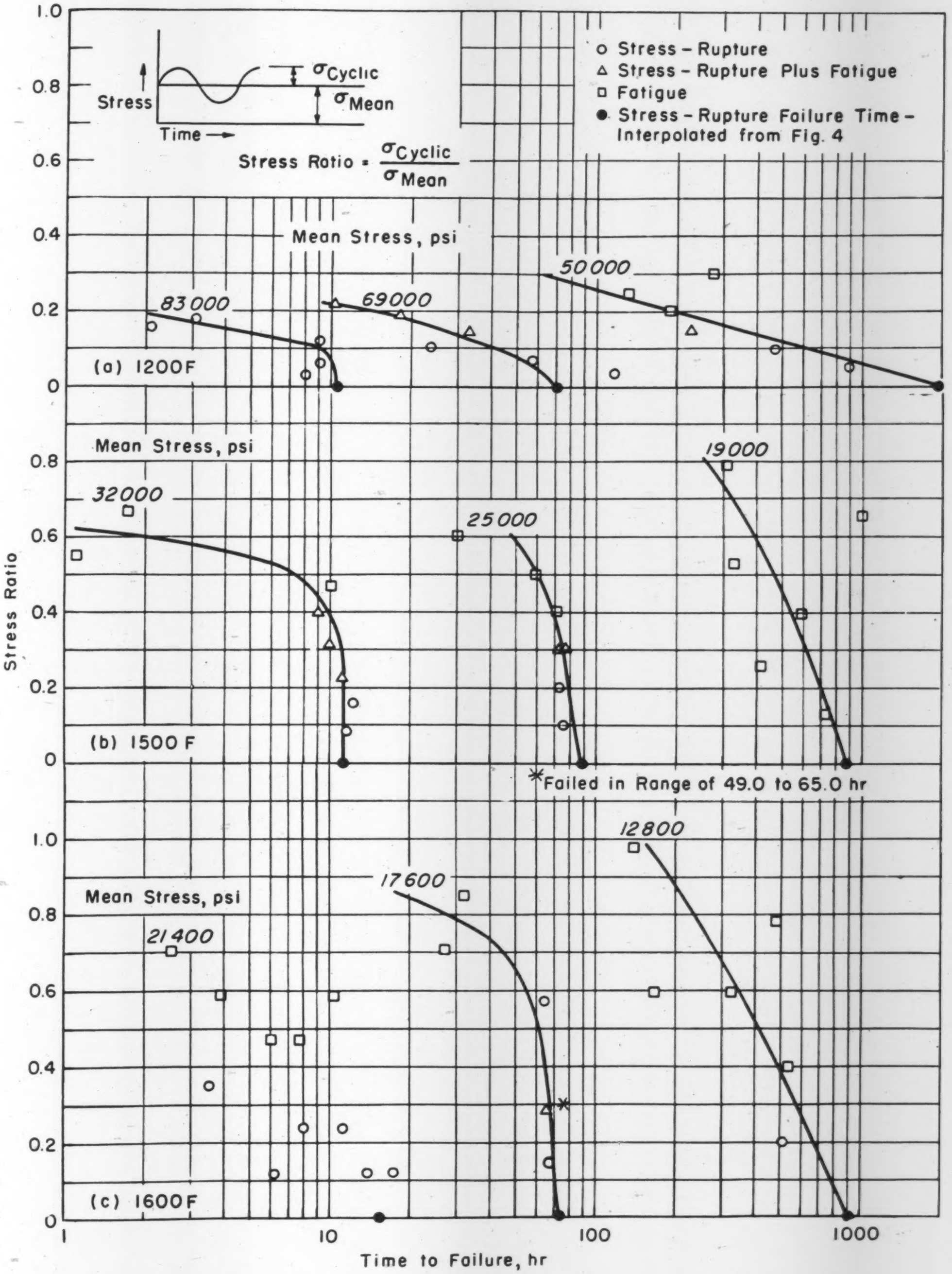

FIG. 5.—Superimposed Relative Cyclic *versus* Life at Constant Mean Tensile Stress.

RESULTS AND DISCUSSION

Fatigue Evaluation:

The stress-rupture data are plotted in Fig. 4 and are comparable to data reported in the literature for S-816 alloy.

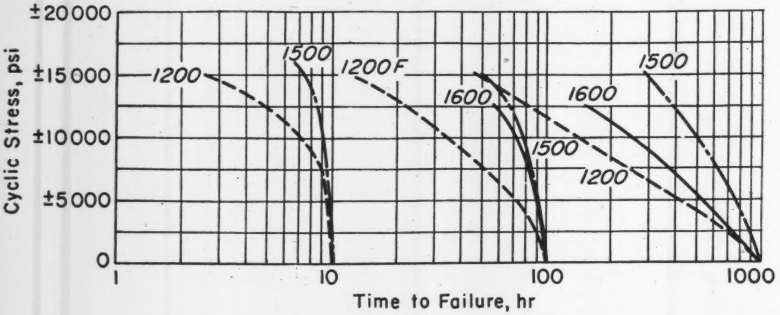

FIG. 6.—Comparison of Effect of Temperature, on Basis of Constant Mean Stress and Absolute Cyclic Stress.

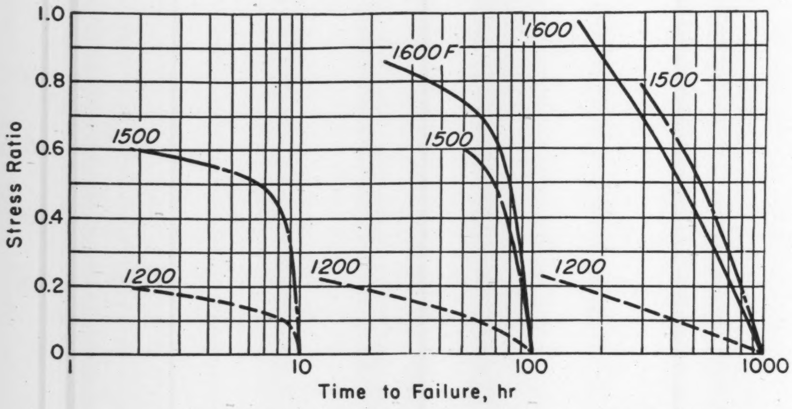

FIG. 7.—Comparison of Effect of Temperature, on Basis of Constant Mean Stress and Relative Cyclic Stress.

Lines were fitted to the data at each temperature by the method of least mean squares. Log-log and semi-log relations between stress and time to failure were considered, but the former was used because it resulted in less scatter.

The results of the fatigue evaluation are summarized in Figs. 5 to 7 in which cyclic stress, on both a relative (that is, stress ratio) and an absolute basis, is related to time to failure at constant mean stress, at each of the temperatures. The stress ratio was defined as the ratio of the amplitude of cyclic to mean stress (see Fig. 5(*a*)). Study of Figs. 5 to 7 indicates the following:

1. In all cases of mean stress and temperature investigated, increasing cyclic stress caused reductions in life (Fig. 5). Under some conditions, however, the reductions in life, if any, were slight at the lower magnitudes of cyclic stress.

2. Superimposed cyclic stresses of given magnitudes (that is, low to intermediate) of stress ratio, Fig. 5, and of absolute stress, Fig. 6, generally resulted in greater relative reductions in life at the lower mean stresses (longer lives)

than at the higher mean stresses (shorter lives); this was so at 1200 and 1500 F and at the two lower mean stresses at 1600 F. The experimental scatter at the highest mean stress (21,400 psi) at 1600 F (Fig. 5) was so extensive that the effect at the highest mean stress is not clear; it is possible to represent a reversal in general trend at this particular condition.

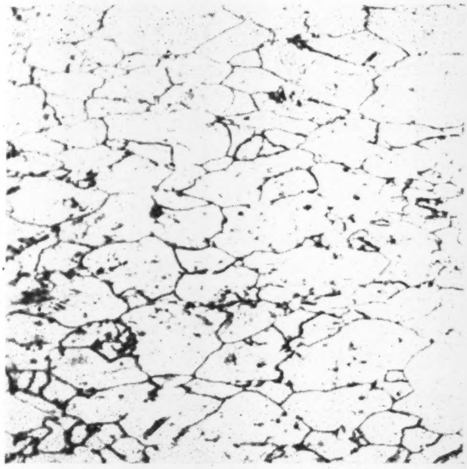

FIG. 8.—Example of Microstructure of Specimens Failed at 1200 F; 65,000 ± 0 psi; 70.0 hr. Electrolytically etched with 5 per cent solution of H_2SO_4 in 90 to 95 per cent boric acid. S-816 (× 250).

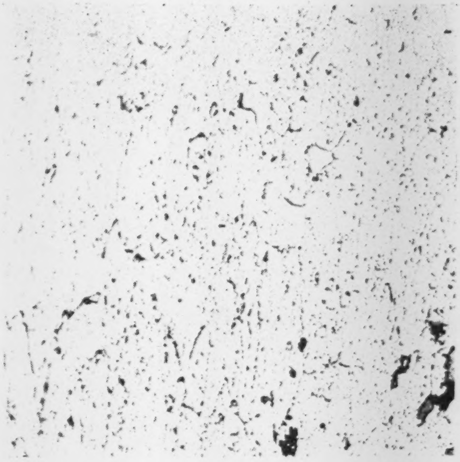

FIG. 9.—Example of Microstructure of Specimens Failed at Zero Vibratory Stress at 1500 and 1600 F; 17,000 ± 0 psi, 1600 F; 101.9 hr. Electrolytically etched with 5 per cent solution of H_2SO_4 in 90 to 95 per cent boric acid. S-816 (× 250).

[NOTE.—In captions of Figs. 8 to 20 the values given after the ± sign are superimposed vibratory stresses.]

3. Based upon equal times to failure at zero vibratory stress, stress ratios of given magnitudes caused greater reductions in life at 1200 F than at 1500 and 1600 F (Fig. 7). The data of Fig. 7 were obtained by adjusting the data of Fig. 5 to give the same nominal times to failure (that is, 10, 100, and 1000 hr) at zero vibratory stress at each of the three temperatures. When cyclic stress is considered on an absolute basis rather than on a relative basis, the same trend is observed but is less pronounced (Fig. 6). This figure was obtained in a similar manner as Fig. 7.

General Metallographic Evaluation:

The structure after operation at 1200 F is illustrated in Fig. 8. Figure 9 illustrates the structure generally associated with specimens having failed at zero vibratory stress at 1500 and 1600 F; elongation of grains will be noted. The structure illustrated in Fig. 10 is generally associated with specimens failing in combined stress at 1500 and 1600 F. Slip planes and twins were observed in the 1200 F specimens, and slip planes and possibly twins in the 1500 and 1600 F specimens. The grain boundaries of the 1500 and 1600 F specimens were generally less distinguishable than those of the 1200 F specimens, indicating perhaps some agglomeration of grain boundary constituent at the two higher temperatures. Slipping and twinning occurred on more than one set of crystallographic planes at all conditions.

There is indication that intergranular corrosion can penetrate considerable distances into AMS 5765A alloy, partic-

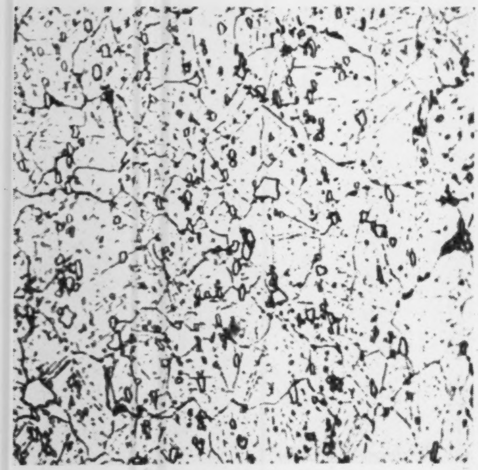

FIG. 10.—Example of Microstructure of Specimens Failed in Combined Stress at 1500 and 1600 F; 17,600 ± 2500 psi, 1600 F; 64.5 hr. Electrolytically etched with 5 per cent solution of H_2SO_4 in 90 to 95 per cent boric acid. S-816 (× 250).

ularly at 1500 and 1600 F, as illustrated in Fig. 11. Presumably this corrosion could be detrimental to both stress-rupture and fatigue strength.

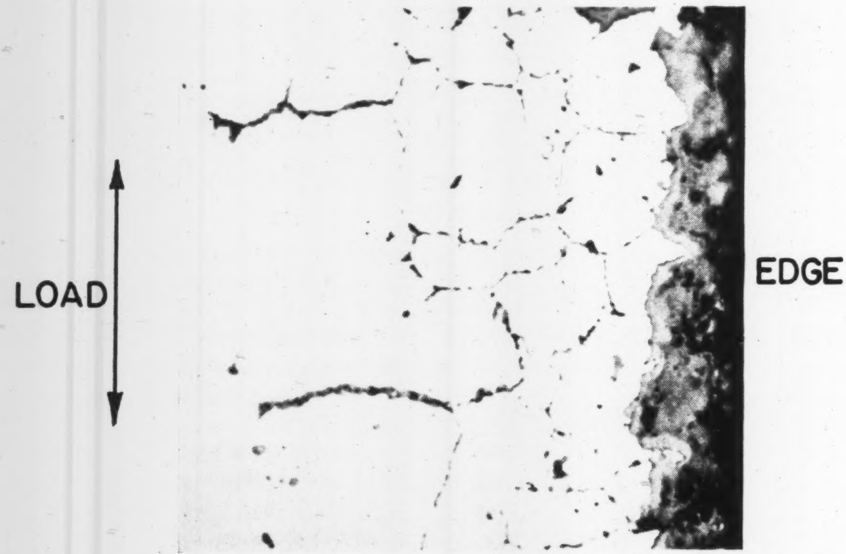

FIG. 11.—An Example of Intergranular Corrosion at 1500 and 1600 F, Along Edge of Specimen; 12,800 ± 12,500 psi, 1600 F; 139.4 hr. Unetched. S-816 (× 250).

Metallurgical Evaluation of Failures:

The summary of the macroscopic study is incorporated in Fig. 5 where each specimen is classified according to type of failure. At 1200 F the stress-rupture failures appeared brittle (Fig. 12), and at 1500 and 1600 F they appeared ductile (Fig. 13). Figure 5 indicates that as the magnitude of superimposed cyclic stress is increased, a transition from stress-rupture to stress-rupture-plus-fatigue failure occurred at all three temperatures. A stress-rupture-plus-fatigue failure is illustrated in Fig. 14. The stress-rupture-plus-fatigue failures were brittle at 1200 F and comparatively ductile at 1500 and 1600 F. Further increases in cyclic stress level then generally resulted in fatigue failure. This type of failure is shown in Fig. 15. These failures were brittle at all temperatures.

Microscopically, the specimens having stress-rupture-appearing failures at 1200

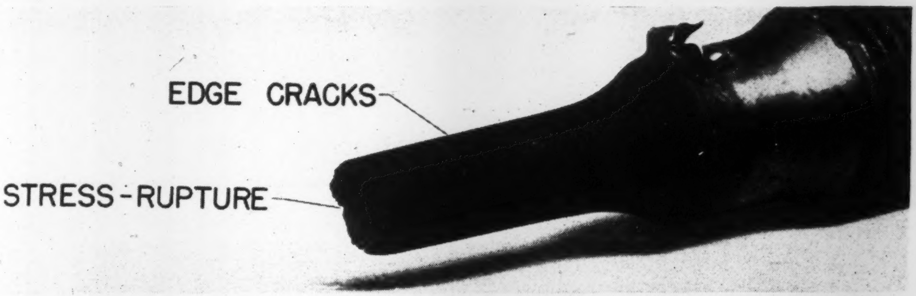

FIG. 12.—Example of Stress-Rupture Failure at 1200 F; 84,000 ± 0 psi; 15.8 hr. S-816 (× 3.5).

F exhibited transgranular shear and cleavage as shown in Fig. 16. The edges of these specimens exhibited transgranular and intergranular cracks (Fig. 17). The specimens having stress-rupture-appearing failures at 1500 and 1600 F generally exhibited transgranular-shear fracture (Fig. 18) and intergranular fracture near the edges (Fig. 19).

The stress-rupture fracture areas in the stress-rupture plus fatigue failures generally appeared microscopically similar to those occurring in the respective stress-rupture-appearing failures at 1200, 1500, and 1600 F.

The fatigue areas fractured transgranularly at all three temperatures. The fracture paths were essentially normal to the applied stress and were, in general, essentially straight lines and did not vary appreciably in direction from grain to grain; this lack of orientation effect from grain to grain gave the appearance of noncrystallinity (Fig. 20). In contrast, as noted, the stress-rupture fractures had been either intergranular or, if transgranular, they followed distinctly differing paths through the grains, indicating shear or cleavage. The surface cracking which occurred at all three temperatures as well as the previously noted corrosion could possibly have nucleated fatigue fracture.

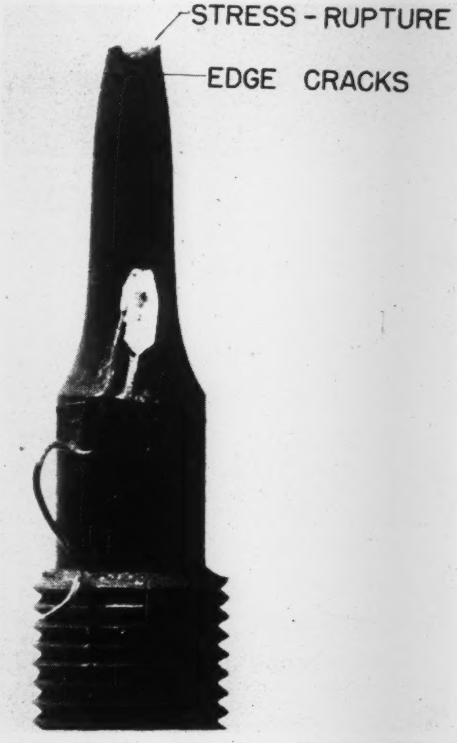

FIG. 13.—Example of Stress-Rupture Failure at 1500 and 1600 F; 19,000 ± 0 psi, 1500 F; 915.4 hr. S-816 (× 3.5).

Relations Between Mean Stress, Cyclic Stress, Temperature, Time to Failure, and Failure Appearance:

A study of Fig. 5 indicates that for all temperatures studied, both fatigue failure and stress-rupture plus fatigue-failure generally were accompanied by reductions in time to failure because of superimposed cyclic stresses. Consequently, if service blades of this alloy, with the same heat treatment, give lives less than expected on the basis of stress-rupture data and simultaneously exhibit fatigue-type failures, vibratory stress would probably be a factor in causing the lowered life. A study of Fig. 5 also indicates that at all temperatures investigated, stress - rupture - appearing failures accompanied lowered life under some conditions of stress. Therefore, blades of this alloy could yield lowered lives because of vibratory stress and yet appear to have failed by stress-rupture alone.

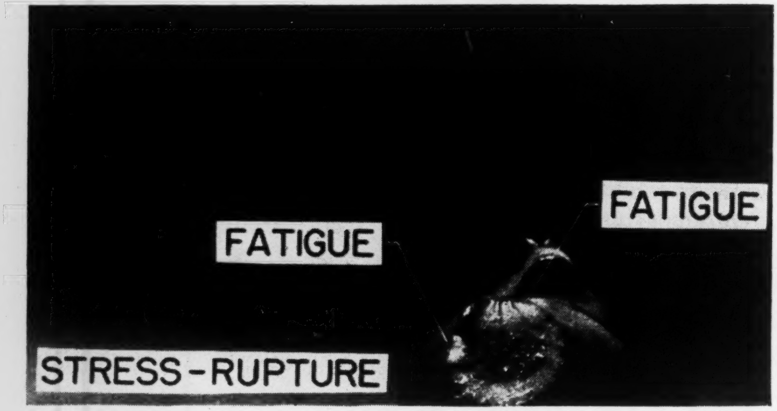

FIG. 14.—Example of Stress-Rupture Plus Fatigue Failure at All Temperatures; 19,000 ± 5000 psi, 1500 F; 415.5 hr. S-816 (× 2.5).

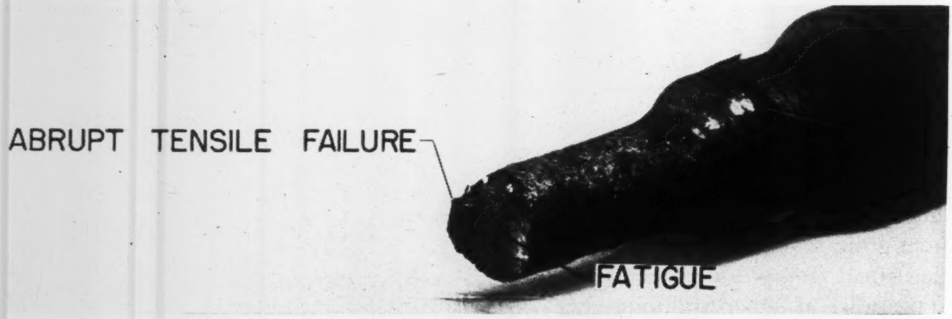

FIG. 15.—Example of Fatigue Failure at All Temperatures; 19,000 ± 2500 psi, 1500 F; 724.4 hr. S-816 (× 3.5).

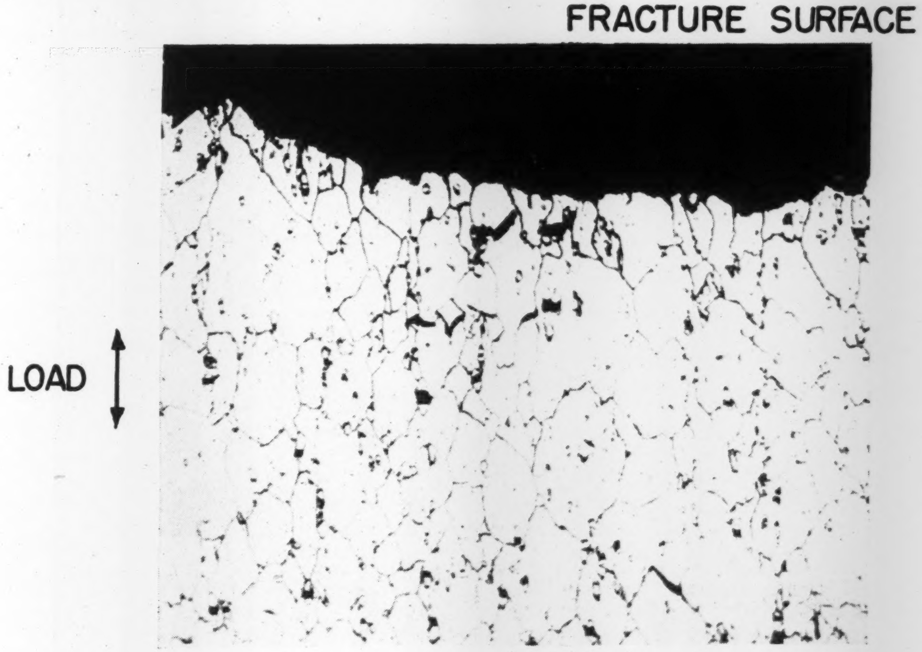

FIG. 16.—Example of Shear and Cleavage Fracture in Stress-Rupture Appearing Failure at 1200 F; 83,000 ± 5000 psi; 9.1 hr. Electrolytically etched with 5 per cent solution of H_2SO_4 in 90 to 95 per cent boric acid. S-816 (× 250).

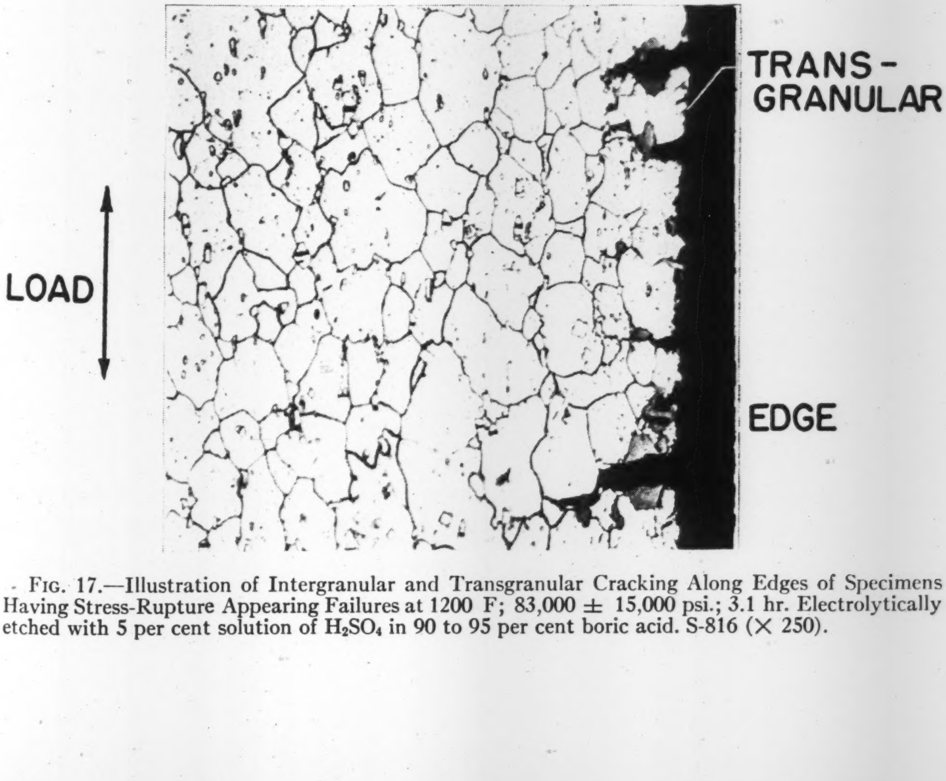

FIG. 17.—Illustration of Intergranular and Transgranular Cracking Along Edges of Specimens Having Stress-Rupture Appearing Failures at 1200 F; 83,000 ± 15,000 psi.; 3.1 hr. Electrolytically etched with 5 per cent solution of H_2SO_4 in 90 to 95 per cent boric acid. S-816 (× 250).

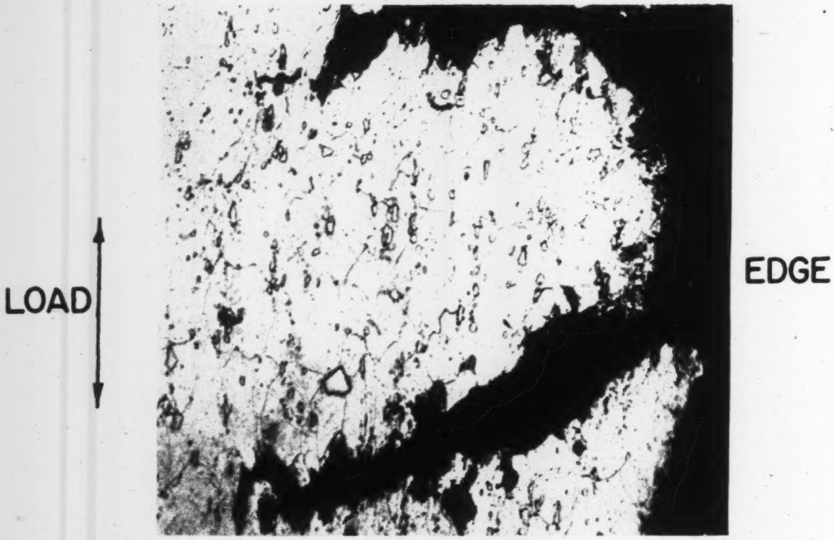

FIG. 18.—Example of Transgranular Shear Fracture in Stress-Rupture Appearing Failures at 1500 and 1600 F; 32,000 ± 2500 psi, 1500 F; 11.5 hr. Electrolytically etched with 5 per cent solution of H_2SO_4 in 90 to 95 per cent boric acid. S-816 (× 250).

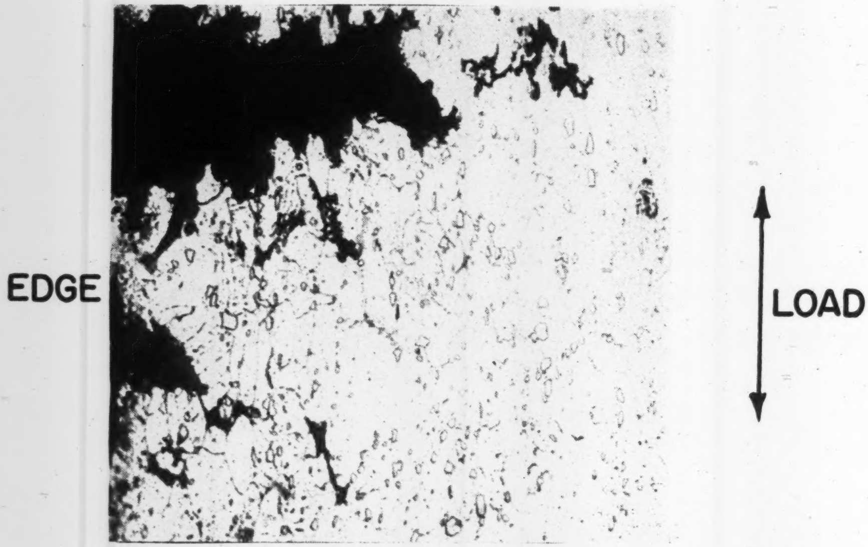

FIG. 19.—Illustration of Intergranular Cracking Along Edges of Specimens Having Stress-Rupture Appearing Failures at 1500 and 1600 F; 21,000 ± 0 psi, 1600 F; 11.4 hr. Electrolytically etched with 5 per cent solution of H_2SO_4 in 90 to 95 per cent boric acid. S-816 (× 250).

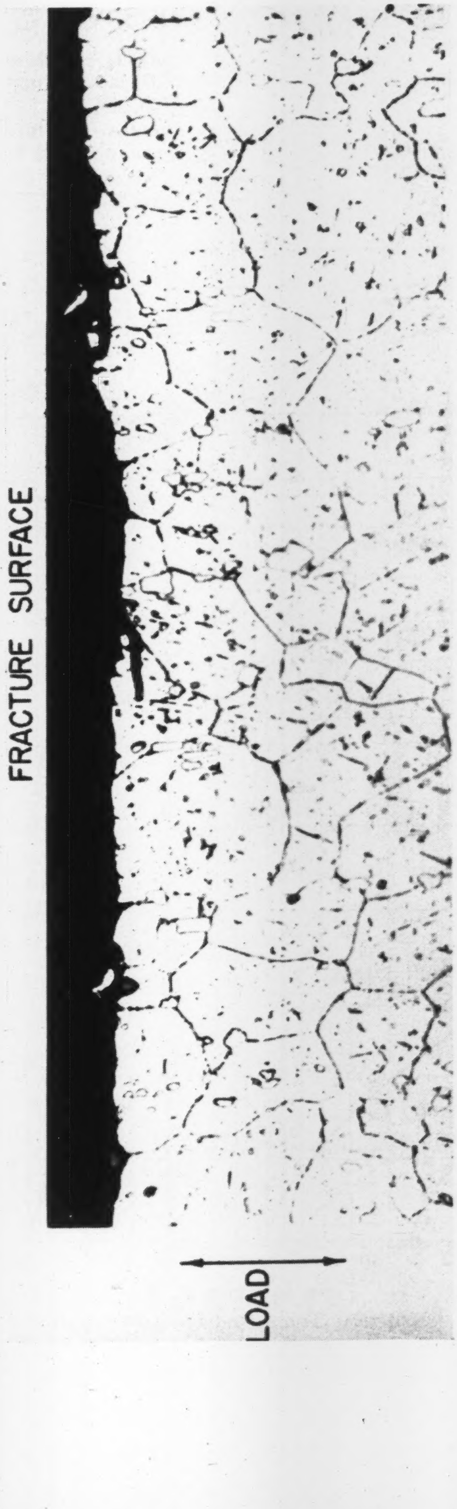

FIG. 20.—Illustration of Fatigue Fracture; 12,800 ± 12,500, 1600 F; 139.4 hr. Electrolytically etched with 5 per cent solution of H_2SO_4 in 90 to 95 per cent boric acid. S-816 (× 750).

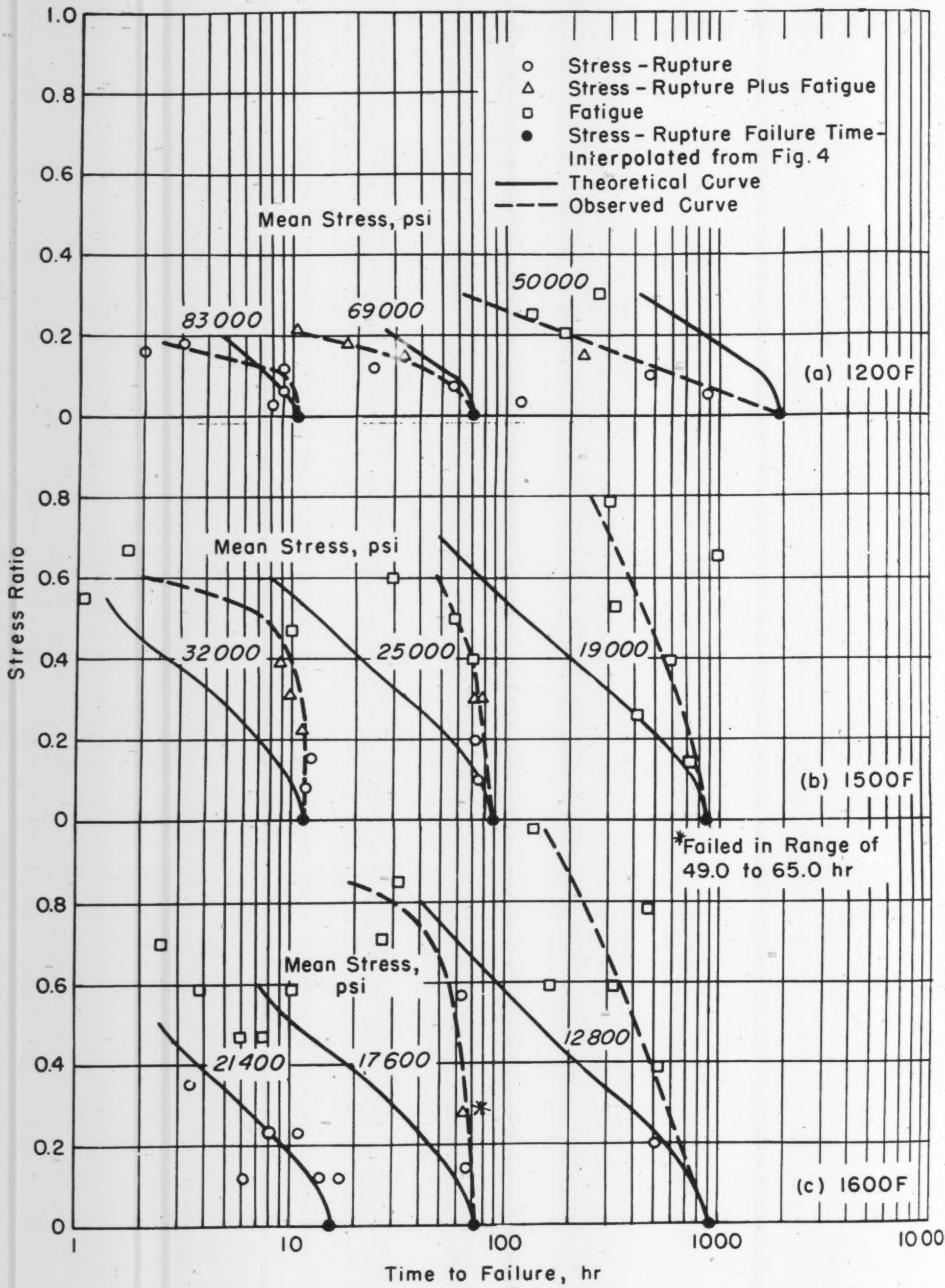

FIG. 21.—Comparison of Theoretical Times to Failure by Stress-Rupture During Dynamic Stressing and Observed Times to Failure.

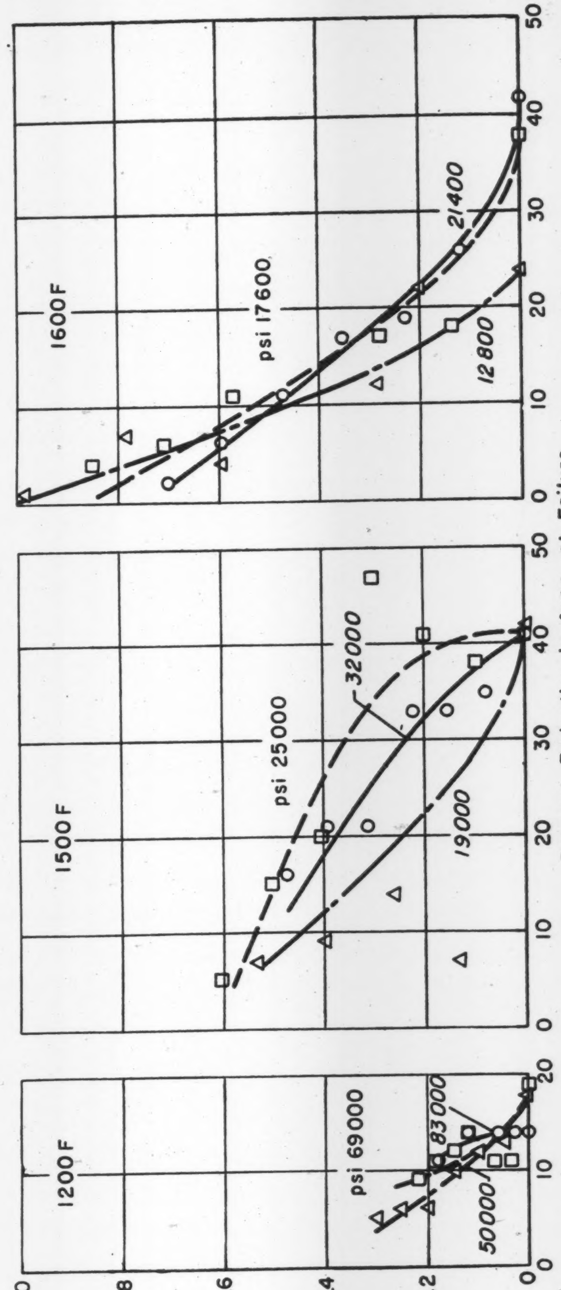

FIG. 22.—Relation Between Stress Ratio and Reduction of Area at Failure.

Comparisons of Observed Times to Failure by Stress-Rupture Under Cyclic Stress Conditions, and Calculated Times to Failure:

Vibratory stress caused reduced time to failure and was associated with stress-rupture-appearing failure at some conditions, as previously noted. Therefore, it became of interest to calculate the time to stress-rupture failure under cyclic stress conditions for comparison with observed times. The assumptions made were that the amounts of stress-rupture damage at the various stress levels during a stress cycle were directly proportional to the times at these stresses and were additive. An assumption of cumulative damage was applied by Miner (2) to the case of fatigue at room temperature. Using standard fatigue data obtained from specimens, each run at a given vibratory stress condition, he predicted failure times for Alclad 2024-T (Alclad 24S-T) aluminum sheet specimens, each run at varying vibratory stress conditions. Quite good agreement was reported on the average. The present assumption of cumulative stress-rupture damage does not consider the possible effects of strain and temperature-dependent metallurgical phenomena; however, this assumption provides an interesting first approach to this problem.

An equation relating time-to-fracture by stress-rupture to mean stress and stress ratio (based upon a constant load stress-rupture curve) is derived in the Appendix. By the insertion of constants from the stress-rupture curves obtained at each temperature (Fig. 4), the time to failure with increased vibratory stress for each mean stress and temperature level has been determined. The hypothetical curves are plotted in Fig. 21 along with the actual data obtained in this investigation.

TABLE I.—HARDNESS AT FAILURE.

Magnitude of Cyclic Stress, σ_{cy}, psi	Rockwell Hardness, A Scale, R_A								
	1200 F			1500 F			1600 F		
	Mean Cyclic Stress, σ_m, psi								
	83 000	69 000	50 000	32 000	25 000	19 000	21 400	17 600	12 800
0.....	71.0	71.5	71.5	68.5	68.5	66.0	65.5	66.0	65.0
2 500.....	72.0	72.0	71.0	68.0	67.0	65.5	64.5	64.5	63.0
5 000.....	72.5	71.5	70.0	68.0	69.0	65.0	66.0	66.0	63.0
7 500.....	72.0	71.5	69.0	68.5	68.0	66.0	65.0	[a]	64.0
10 000.....	72.5	71.0	69.0	67.0	68.0	65.0	64.5	65.5	68.5
12 500.....	...	70.5	68.5	68.5	66.5	[a]	65.5	64.0	64.0
15 000.....	70.0	70.0	69.0	68.5	64.5	70.0	62.0	64.0	...
21 440.....	...	...	...	66.0	...	...	...	...	...

[a] Specimen misplaced after testing.

Although close agreement between the hypothetical curves and experimental data might not be expected under high vibratory stress which clearly introduced fatigue characteristics into the failure, the hypothetical curves have been extended to these values to allow comparisons to be made up to the maximum stress ratios studied herein.

The hypothetical curves do predict the observed decrease in life with increased stress ratio; the agreement with the data is not good, however. At the low temperature (1200 F) vibratory stress (fatigue) is more damaging, and at high temperatures (1500 and 1600 F) less damaging than predicted by the cumulative damage theory.

Reduction of Area at Failure.—The percentage reduction in area at failure for the various conditions studied are presented in Fig. 22. Study of this figure reveals that the reduction of area, at failure, decreased with increased cyclic stress. For example, at 1600 F the reductions in area at zero vibratory stress were of the order of 25 to 40 per cent but less than 5 per cent with stress ratios of about 0.8. Elongation would be expected to have similar trends.

Hardness at Failure.—The hardnesses at failure are presented in Table I. There is no highly pronounced trend between the hardness at failure and cyclic stress, though there is slight indication of lower hardness at failure at the higher vibratory stresses.

SUMMARY OF RESULTS

The results of an investigation of the relations between superimposed cyclic stress, mean stress, temperature, time to failure, and failure characteristics of wrought S-816 alloy are as follows:

1. Increasing cyclic stress reduced time to failure (life) at all conditions of mean stress and temperature investigated. Under some conditions, however, the reductions, if any, were slight at the lower cyclic stress magnitudes.

2. Superimposed cyclic stresses of a given magnitude (low to intermediate) generally resulted in relatively greater reductions in life at the lower mean stresses (longer lives) than at the higher mean stresses (shorter lives).

3. Cyclic stresses of given magnitudes caused greater reductions in life at 1200 F than at 1500 and 1600 F.

4. With increased cyclic vibratory stress about a mean stress, the appearance of the failure was indicated as undergoing a transition from that of stress-rupture to a combination of stress-rupture and fatigue. Further increases in vibratory stress level resulted in the occurrence of fatigue failure.

5. The appearance of fatigue-type failures was generally accompanied by reductions in life because of cyclic vibratory stress.

6. Under some conditions, stress-rupture-appearing failures occurred even though the specimen life had been lowered by cyclic stress.

7. Stress-rupture fracture occurred by transgranular cleavage and shear at 1200 F and occurred by intergranular failure and transgranular shear at 1500 and 1600 F. Fatigue fracture was transgranular at all three temperatures and did not appear to follow crystallographic planes.

8. The percentage reduction in area at failure (fracture ductility) decreased with increased vibratory stress at all conditions investigated.

REFERENCES

(1) Robert F. Ferguson, "Effect of Magnitude of Vibratory Load Superimposed Upon Mean Tensile Load on Mechanism of and Time to Fracture of Specimens and Correlation to Engine Blade," United States National Advisory Committee for Aeronautics Research Memorandum E 52I17 (1952).

(2) Milton Miner, "Cumulative Damage in Fatigue," *Journal*, Applied Mechanics, September, 1945, pp. A-159 to A-164.

APPENDIX

DETERMINATION OF TIME TO FAILURE BY STRESS-RUPTURE DURING DYNAMIC LOADING

The development of a hypothetical relation between magnitude of cyclic stress, mean stress, and time to failure by stress-rupture at a given temperature is as follows: The fraction of stress-rupture life consumed at each stress level (that is, stress-rupture damage) during a dynamic stress cycle was assumed to be equal to the ratio of the time at that stress level to the total stress-rupture life at that stress level. Further, the fractions of stress-rupture life consumed at the different stress levels were assumed to be additive, so that the total fraction of stress-rupture life consumed during one cycle of stresses is:

$$\sum_{i=1}^{i=n} \frac{\Delta\theta_i}{\theta_{r,i}}$$

where:

θ_r = time to fail by stress-rupture at a given stress level, hr,

$\Delta\theta$ = duration of time at given stress level, hr, and

n = number of time intervals in a single cycle.

Since the stress varies continuously during a stress cycle, the total fraction of stress-rupture damage per cycle of stresses may be expressed as:

$$\int_0^{2\pi/\omega} \frac{d\theta}{\theta_r}$$

where:

ω = frequency of cyclic stress, radians per hr.

If it is assumed that the static stress-rupture relation applies and is defined by:

$$\theta_r = CS^B (\text{That is, } \log \theta_r = \log C + B \log S)$$

where:

C, B = constants for given temperature and obtained from stress-rupture relation for desired temperature, and

S = stress, psi.

and that:

$$S = S_m(1 + A \sin \omega\theta)$$

where:

S_m = average cycle stress, and

A = ratio of amplitude of cyclic to mean stress.

then:

$$\int_0^{2\pi/\omega} \frac{d\theta}{CS_m^B(1 + A \sin \omega\theta)^B} \times 100 = \frac{2\pi}{\omega} R$$

where:

R = average rate of stress-rupture damage under cyclic stress conditions, per cent per hr.

Finally, the assumption was made that stress-rupture failure occurred when cumulative stress-rupture damage became equal to unity. That is, failure time was equal to the reciprocal of the average damage rate, thus:

$$\theta_e = \frac{100}{R}$$

where:

θ_e = time to failure by stress-rupture under cyclic stress conditions, hr.

The final relation becomes:

$$\int_0^{2\pi/\omega} \frac{d\theta}{S_m^B(1 + A \sin \omega\theta)^B} = \frac{2\pi C}{\omega\theta_e}$$

Graphical integration of the above equation for given conditions, using the appropriate stress-rupture relation from Fig. 4 gives the corresponding values of time to failure. Or the left denominator can be inverted, the binomial expanded, and the resulting series integrated. The value of time to failure may be determined in this way, the accuracy of the answer depending upon the degree of expansion of the series.

A REVIEW OF METHODS EMPLOYED IN THE STATISTICAL ANALYSIS OF FATIGUE DATA*

BY R. ROELOFFS[1] AND F. GAROFALO[2]

Statistical methods have been used successfully in recent years in analyzing the nature of fatigue in metals (**1–3, 5–11, 19, 20**).[3] Many procedures and techniques have been proposed by a number of investigators in treating the many aspects of the fatigue problem. Because the literature on the statistical aspects of fatigue is voluminous and scattered throughout many technical publications, it is desirable to summarize concisely the various techniques which have been proposed and used up to now.

Such a summary which deals primarily with the engineering rather than the theoretical aspect of the problem of fatigue, is presented in this paper in which the fatigue problem is subdivided into four different sections including: (*a*) variability in fatigue life at a fixed stress level; (*b*) determination of endurance limit among nominally identical specimens; (*c*) empirical fitting of *S*-*N* curves, and (*d*) fundamental theories of fatigue failure. Although each of these sections is discussed separately, they are not entirely independent. Also discussed is an "up-and-down" statistical method which has not as yet been used extensively for measuring the endurance limit. Initial results on a manganese-molybdenum steel using this method are given.

VARIABILITY IN FATIGUE LIFE AT FIXED STRESS LEVEL

It has long been recognized that the variability in fatigue life from specimen to specimen tested at the same stress is too large to be accounted for by the ordinary types of experimental error occurring from specimen preparation and testing. With even the most elaborate precautions to insure uniformity, there remains a very large variability which can be attributed only to inherent differences among specimens. This variability is so large that the average of several specimens is usually inadequate to describe the results properly. Instead, it is necessary to consider also the frequency distribution of fatigue life at a given stress.

Comparison Between Logarithmic Normal and Extreme-Value Distribution:

The most thorough investigation of the distribution of fatigue life was by Mueller-Stock as described by Freudenthal (**1**), in which 235 steel specimens were tested at a single stress level. The resulting distribution (Fig. 1(*a*)) was decidedly askew to the right when plotted against N, the number of cycles to failure. However, if plotted in terms of log N, (Fig. 1 (*b*)) the distribution becomes more symmetrical and approximates the normal distribution.

---

* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] Merck and Company, Inc., Rahway, N. J., formerly with United States Steel Corp., Fundamental Research Laboratory, Kearny, N. J.

[2] United States Steel Corp., Research Center, Edgar C. Bain Lab. for Fundamental Research, Monroeville, Pa.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1090.

Other investigators (**1, 2**), on the basis of fewer observations on a variety of materials, have reported good agreement with the normal distribution when plotted on a logarithmic scale. This distribution is generally referred to as the logarithmic normal, or lognormal, distribution.

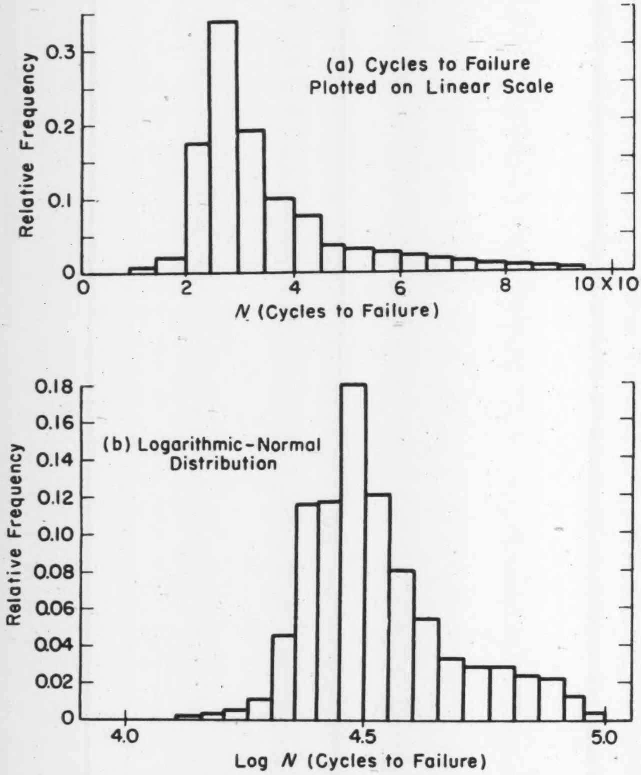

FIG. 1.—Frequency Distribution of Fatigue Life Computed from 235 Tests at Constant Stress (After Freudenthal).

A different distribution, the extreme-value distribution, has been proposed by Gumbel and Freudenthal (**3, 19**) and has been shown by them to fit the data of Ravilly at least as well as the logarithmic normal distribution. One form of the extreme-value distribution is very similar in appearance to the logarithmic normal except in the vicinity of the tails. It is estimated that thousands of tests at the same stress would be required to show a detectable difference between these two distributions experimentally. While there are theoretical reasons for preferring the extreme-value distribution, since it is based on a plausible theory of the nature of fatigue failure, the logarithmic normal distribution is somewhat easier to handle and represents a satisfactory approximation unless interest is centered in the regions of very low probability of failure. It is, therefore, convenient and proper to use logarithmic probability paper for the presentation of results at a fixed stress level, because on this paper the expected proportion, P, of specimens failing plots as a linear function of N, the number of cycles. A value of P is obtained in the following manner for each specimen which fails. If n is the number of specimens tested and the kth failure occurs at N_k cycles, then

$$P_k = \frac{k}{n + 1} \qquad (1)$$

TABLE I.—FATIGUE LIFE OF INGOT IRON WIRE SPECIMENS TESTED IN REVERSED TORSION AT 31 ± KG PER SQ MM BY RAVILLY AS GIVEN BY FREUDENTHAL (**1**).

k	P_k	N_k	log N_k
1	0.048	11.2×10^3	4.049
2	0.095	11.7	4.068
3	0.143	11.8	4.072
4	0.190	11.9	4.076
5	0.238	12.0	4.090
6	0.286	12.3	4.990
7	0.333	12.4	4.094
8	0.381	12.8	4.108
9	0.429	13.1	4.116
10	0.476	13.1	4.116
11	0.524	13.3	4.124
12	0.571	13.4	4.128
13	0.619	13.4	4.128
14	0.667	13.5	4.131
15	0.714	13.7	4.137
16	0.762	13.8	4.140
17	0.810	13.8	4.140
18	0.857	14.0	4.147
19	0.905	14.1	4.150
20	0.952	14.6	4.165

gives the proportion, P_k, of failures to be plotted at N_k cycles. Dividing by $n + 1$, rather than by n, gives a better estimate of the proportion of specimens that will fail prior to N_k cycles. That this is reasonable may be seen by considering a test of an odd number of specimens. If the observed lives are arranged in order of magnitude, the middle value should provide an estimate of the median life and should therefore be plotted with $P_k = 0.5$, which is the value that will be obtained from Eq 1 for the middle specimen in a set containing an odd number of specimens.

Comparison with Experimental Results:

As a numerical example, the data in Table I, from Ravilly (**21**) for ingot iron wire tested in torsion, are used. The calculated values of P are plotted in Fig. 2 against the corresponding values of log N. Plotting log N on probability paper is equivalent to plotting N on logarithmic probability paper, but permits greater freedom in choice of scale. The straight line drawn through the points was obtained by calculating the mean and the standard deviation of the log N values. Since the normal distribution is symmetrical, and for a symmetrical distribution the mean and the median coincide, the mean of the log N values gives the point where the line intersects the 50 per cent failure line. Also for a normal distribution, 84 per cent of the failures occur prior to the mean plus 1 σ, so that the slope of the line through the observed points can be determined. It is also possible to reverse the procedure and draw the line by eye and then use the fitted line to estimate the mean life and standard deviation. Ordinarily, it would be preferable to calculate the values, because the element of subjectivity in fitting the line is unde-

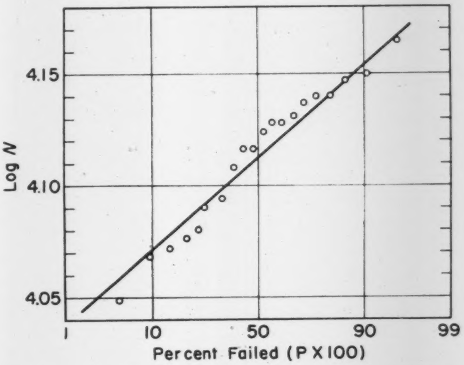

FIG. 2.—Fatigue Life in Ingot Iron Wire Specimens Tested in Reverse Torsion at ± 31 kg per sq mm by Ravilly (**21**).

sirable. There is a commonly occurring situation in fatigue testing, however, in which the graphical method has an advantage. When testing at a stress level near the endurance limit it is frequently impractical to continue testing until all the specimens have failed. The test is then stopped without obtaining the life values for several of the specimens. Application of Eq 1 permits plotting the points for those specimens which failed in exactly the same manner as if the test had been continued until all specimens failed. The only effect is to remove those points at the upper right end of the curve which correspond to the unfractured specimens. Drawing a line through the known points will still permit estimating the mean and standard deviation of the entire population, even though ordinary methods of calculation fail. Actually, analytic methods are also available for this situation, but they are no longer simple (**4**).

Methods for Truncated or Censored Data:

The method of dealing with the surviving specimens when testing at a constant stress is a cause of some disagreement in the literature. Several authors have analyzed such data by merely ignoring the surviving specimens and calculating the average and standard deviation of the fractured specimens (**5, 6, 7, 8, 9**). Two justifications for the procedure are given. First, it is stated that there is no way of including the surviving specimens. Actually the method described above can be used. Second, one can consider that the surviving specimens belong to a different population from the fractured ones, and interest is then centered first on the proportion of surviving specimens, which are presumed to have infinite life, and next on the average and standard deviation of the fractured specimens, which are presumed to be a sample from the population of specimens with finite life. If this viewpoint is accepted, then ignoring the surviving specimens when estimating the mean and standard deviation of the population of specimens with finite life is proper. It seems more satisfactory, however, to consider all specimens as belonging to the same population, and that some of the specimens survive only because it is impractical to wait for the fracture of the very long-lived specimens. This point of view implies using all the specimens in the calculation in the manner described previously. The censored frequency distribution technique has been employed recently (**23**) in testing aluminum alloys, which do not show a fatigue limit.

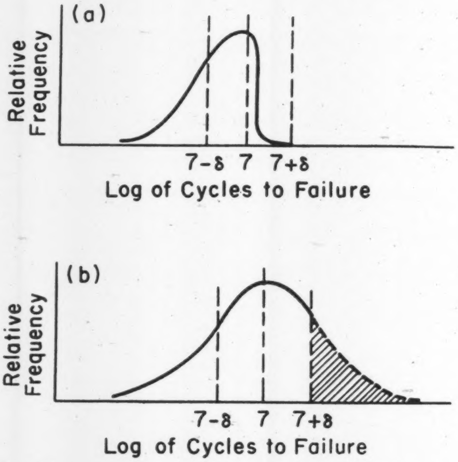

FIG. 3.—Fatigue Life Distributions of Tests at Constant Stress Assuming (*a*) that All Surviving Samples Belong to a Population with Infinite Life and (*b*) that All Samples Belong to the Same Population.

The question of which method is appropriate could be settled on experimental grounds by testing a fairly large number of specimens at a fixed stress level chosen so that about half the specimens survive 10^7 cycles. If the test is run for a number of cycles beyond 10^7, say to $10^{7+\delta}$ cycles, then the point of view that specimens which survive 10^7 cycles belong to a different population with infinite life implies a finite life distribution of the type shown in Fig. 3(*a*). There, the number of specimens failing in the interval 7 to $7 + \delta$ is substantially less than the number failing in the interval $7 - \delta$ to 7. On the other hand, the view that all specimens are from the same population implies a life distribution like that of Fig. 3 (*b*), in which the number of failures in the interval $7 + \delta$ is roughly equal to the number of failures in the interval $7 - \delta$ to 7.

DETERMINATION OF ENDURANCE LIMIT

An important aspect of fatigue testing is the determination of the endurance limit in materials that exhibit this phenomenon. A generally accepted point of view is that each specimen has its own endurance limit, a stress level above which it will fail after a finite number of cycles and below which it will survive for a very large number of cycles. Because the extremely long-lived specimens cannot be tested for indefinitely long periods of time to reach fracture, it is necessary to redefine the endurance limit in terms of some specified number of cycles. The critical value of stress is then usually referred to as the fatigue strength for the specified number of cycles.

Unfortunately, it is impossible to determine the endurance limit for a single specimen. If a specimen is tested at a fixed stress, the only information obtained is whether the test stress is above or below the endurance limit for that specimen. A method described by Stulen (**10**) could be used in principle to determine the endurance limit of a single specimen, but for several practical reasons cannot be applied. Nevertheless, this method is worth considering, in order to define more clearly the concept of the endurance limit. A specimen is tested at a low stress level. If it survives a predetermined number of cycles, the test is repeated on the same specimen at a slightly higher stress level. This is continued until failure occurs. The highest stress at which the specimen survives is then taken as the estimated endurance limit. This would be a tedious procedure and it cannot always be assumed that the specimen is unaffected by testing below its endurance limit.

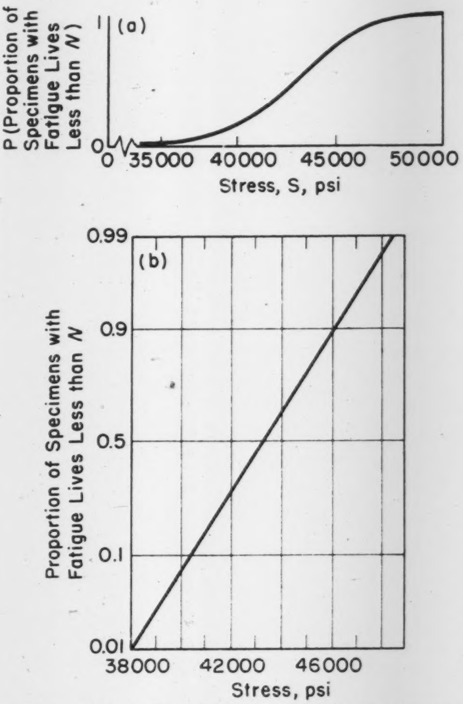

FIG. 4(*a*).—The Proportion of Specimens with Fatigue Lives Less than *N* and (*b*)—The Results in (*a*) Plotted on Normal Probability Paper.

Determination of Median Endurance Limit by Probit Analysis:

In reality the endurance limit of a particular specimen is not of much interest from the viewpoint of engineering design, since experimental results indicate a considerable variation in the endurance limit of individual, nominally identical specimens. Interest, from this point of view, is therefore centered on the standard deviation or confidence limits that can be imposed on the median endurance limit. The latter is usually defined (8) as the stress where 50 per cent of the samples fail within a predetermined number of cycles.

In determining experimentally (8) the median endurance limit, a predetermined number of specimens are tested at various given stress levels, which presumably bracket the endurance limit. Results of this type show the existence of approximately a normal distribution of fatigue life (5, 7–9). In plotting the portion of the specimen with fatigue lives less than a fatigue life N against the stress, as done schematically in Fig. 4(*a*), a sigmoid curve is found experimentally (8). For an essentially normal distribution of the fatigue life, the results of Fig. 4(*a*), as shown in Fig. 4(*b*), plot as a straight line on probability paper. As defined previously, the median fatigue limit is then obtained from Fig. 4(*b*), by determining the stress corresponding to a portion of failed specimens of 0.5. The standard deviation is then determined from the slope of the straight line in Fig. 4(*b*) following a probit transformation (22).

It should be pointed out that in discussing the probit analysis of the median endurance limit, the terminology employed will cause confusion if it implies an experimentally determined distribution of fatigue limits. Although such a distribution of fatigue limits may exist, it has not been determined as yet.

Determination of Median Endurance Limit by the "Up-and-Down" Method:

A somewhat different approach to the estimation of the median endurance limit is the "up-and-down" method (5, 11, 12, 13). Whereas in probit analysis the number of specimens tested at each stress level must be determined in advance, in the "up-and-down" method the stress used to test each specimen is based on the results of the preceding test. It is thus possible to concentrate the observations more closely about the median endurance limit. The stress at which the first specimen is tested is chosen as close to the expected median endurance limit as is possible from prior knowledge or by testing a sample using the method described by Stulen (10) but omitting this test from the analysis described below. An increment of stress, called the step size, is also chosen in advance. If the first specimen survives the test for a fixed N, the stress is increased by one step for the second specimen. If the first specimen fails, the stress is decreased by one step for the second specimen. Each succeeding specimen is tested at a stress level either one step higher or lower than the preceding specimen, as determined by the above rule. Each specimen thus provides a stress value at which to test the next specimen. The average of these stress values is an estimate of the median endurance limit. This procedure can be considered to give an estimate of the median endurance limit which is as precise as one obtained by probit analysis, but requiring 30 to 40 per cent fewer specimens (12).

Results on a Manganese-Molybdenum Steel Using the "Up-and-Down" Method:

A slight disadvantage of the up-and-down method is that only one specimen can be tested at a time, since the results of the preceding test are necessary to determine the stress level for the next specimen. It is possible, however, to run separate up-and-down sequences on several machines. The machine averages can then be compared, and combined to give one grand average. This method was used to estimate the endurance limit of a manganese-molybdenum steel containing 0.49 per cent carbon, 1.53 per cent manganese and 0.35 per cent molybdenum.

The rotating-beam fatigue specimens tested were machined from blanks $3\frac{7}{16}$ in. long and $\frac{1}{2}$ in. in diameter. The machined specimens were heat treated before final grinding and polishing. Except for a few modifications the machining, grinding, and polishing techniques in preparing the fatigue specimens are the same as those described previously (**17**). The finished specimens had a $1\frac{1}{2}$ in. long, 10-in. radius reduced section 0.200 in. in minimum diameter. The test specimens were heat treated by austenitizing at 1550 F for $1\frac{1}{2}$ hr, quenched in oil and tempered for 20 hr at 1230 F. This treatment resulted in a spheroidized structure with a Vickers hardness of 223 (19 Rockwell C) and a tensile strength of 103,000 psi.

Twenty specimens were tested on four R. R. Moore type rotating-beam fatigue machines. Five specimens were assigned to each machine and a step size of 1000 psi was chosen. A step size equal to the standard deviation of the distribution of fatigue limits is the optimum choice, but because this number is not known in advance, it is necessary to guess at an appropriate step size. Fortunately, the procedure is not very sensitive to moderate departures from the optimum choice, and a value anywhere from one half the standard deviation, σ, to $2\ \sigma$ is satisfactory.

In selecting the initial stress level for the up-and-down method it is generally not possible to select with any certainty a stress within one or two step sizes of the median endurance limit. Thus, unless precautions are taken, there may be a long run of either fractured or surviving specimens before the neighborhood of the median value is reached. This is especially true if the step size happens to be small. It has been suggested (**13**) that such initial runs be omitted from the data and computations made on basis of fewest events. In this investigation the initial stress level was determined by the Stulen procedure which is found to give a satisfactory estimate of the level of the median endurance limit for the material tested. The chance of having a long run of either fractured or surviving specimens is therefore minimized.

TABLE II.—RESULTS OF FATIGUE TESTS ON SPECIMENS OF MANGANESE-MOLYBDENUM STEEL.

Machine	Stress (1000 psi)	Result[a]
I	59	F 1
	58	S 0
	59	S 0
	60	S 0
	61	F 1
II	61	F 1
	60	S 0
	61	F 1
	60	S 0
	61	F 1
III	59	S 0
	60	S 0
	61	F 1
	60	F 1
	59	F 1
IV	58	S 0
	59	S 0
	60	F 1
	59	F 1
	58	S 0

[a] F denotes failure in less than 10^7 cycles, and S denotes survival beyond 10^7 cycles.

The test results obtained by using the up-and-down method are given in Table II. In this table, F denotes failure in less than 10^7 cycles and S denotes survival to at least 10^7 cycles.

Estimate of Median Endurance Limit:

The analysis of the results in Table II follows the procedure described by Dixon and Mood (**12**). A shortened version of their paper also appears in a book by

Dixon and Massey (14). The analysis is based on either the survivals or the failures, whichever has the smaller total. Although for the data in Table II, there are ten of each, the failures are used to represent "success" events in the Dixon and Mood analysis. The total number of failures is denoted by N, and n_0, n_1, and n_2 represent the number of failures at each level of stress, with n_0 $(=59)$ the number of failures at the lowest level at which failures occur. We have then

$$\sum_{i=0}^{2} n_i = 3 + 2 + 5 = 10 = N \quad (2)$$

The quantities A and B are calculated from

$$A = \sum in_i = 12$$
$$B = \sum i^2 n_i = 22 \quad (3)$$

The estimated median endurance limit, m, is then given by

$$m = y' + d\left[\frac{A}{N} \pm \frac{1}{2}\right] \quad (4)$$

where y' is the stress level corresponding to the lowest level at which failures occur, and d is the step size. The plus sign is used when the analysis is based on surviving specimens, the minus sign when it is based on failures. Thus the value of m is obtained as follows:

$$m = 59{,}000 + 1000(1.2 - 0.5) = 59{,}700 \text{ psi} \quad (5)$$

The standard deviation, σ, of the endurance limits of individual specimens is estimated by s, where

$$s = 1.620d\left[\frac{NB - A^2}{N^2} + 0.029\right] = 1278 \text{ psi} \quad (6)$$

In order to obtain confidence intervals on the estimated median fatigue limit, the standard deviation s_m of the estimated endurance limit is required. This is calculated from

$$s_m = \frac{G_s}{\sqrt{N}} \quad (7)$$

which corresponds to the usual formula for the standard deviation of a mean of a sample of N observations, except for the additional factor G. This factor depends on the ratio d/σ, and is charted by both Dixon and Mood (12), and Dixon and Massey (14). Because d/σ is unknown, the chart is entered using the estimated value, d/s.

For our results

$$s_m = \frac{(0.96)(1278)}{\sqrt{10}} = 388 \text{ psi} \quad (8)$$

An α per cent confidence interval is given by

$$m \pm t(\alpha, N - 1)s_m, \quad (9)$$

where $t(\alpha, N - 1)$ is the α per cent point of Student's "t" distribution with $N - 1$ degrees of freedom. For the results in Table II, the median endurance limit with a 95 per cent confidence interval is

$$m = 59{,}700 \pm (2.26)(388) = 59{,}700 \pm 877 \text{ psi} \quad (10)$$

EMPIRICAL FITTING OF S-N CURVES

Several empirical functions have been proposed which fit observed fatigue data quite well (15, 16, 19). It has been pointed out by several investigators (3, 16) that it is misleading to speak of the S-N curve as if there were only one such curve. Actually, there is an S-N curve defined for each probability of failure, the curve for 0.5 probability being the one that in engineering practice is generally referred to as the S-N curve. The S-N curve for a probability greater than 0.5 should be shifted to higher imposed stresses, whereas for a probability less than 0.5 it should be shifted to lower stresses. If it were possible to establish the existence of a distribution of fatigue limits for different fatigue lives, the interrelationship between the fatigue life, stress, and probability of failure could be readily shown.

Of the empirical relations between stress and fatigue life, Weibull has proposed two which cover fatigue life from $N = 1$ to $N = \infty$. These relationships are:

$$S - S_o = A(N + B)^{-m} \ldots\ldots (11)$$

where
S_0 = endurance limit, and
A, B, and m = parameters to be estimated from the data, and

$$\frac{S - S_o}{TS - S_o} = \text{Exp}\left[-k(\log N)^m\right] \ldots (12)$$

where TS = tensile strength, and k and m are other parameters to be estimated from the data.

A third function proposed by Weibull is designed to fit only the lower portion of the curve in the region of the endurance limit and is given as

$$N = A(S - S_o)^{-m} \ldots\ldots\ldots (13)$$

By appropriate transformation it is possible to represent these functions by straight lines. In each case there is a different line (or a different set of parameters) for each specified probability of failure. The computations necessary to determine the constants become extremely difficult unless a number of specimens are run at each of several stresses. There must be at least as many stresses used as there are parameters to be estimated in the chosen function. To obtain reasonable estimates of the S-N curves for high and low probabilities of failure requires more specimens at each stress level than estimating only the median ($P = 0.50$) curve. It would appear that the endurance limit obtained by this method will be less precise for the same number of specimens than an estimate obtained using the probit or staircase methods, which tend to concentrate the observations in the region where more information about the endurance limit is obtained per observation. However, the S-N curves give some information about behavior at higher stresses, which is not obtained by the probit or staircase methods.

FUNDAMENTAL THEORIES OF FATIGUE

Several theories of fatigue fracture based on statistical distribution functions have been proposed. These consider that the stressed material is composed of a large number of small elements of volume. Due to the inhomogeneity of the material, each element has a different strength. This is due to the presence of crystal imperfections, inclusions, and various other submicroscopic flaws, all of which may vary in size, shape, and orientation. As a result, the strengths of the elemental volumes have a frequency distribution and the elements of volume in the critically stressed region of a fatigue specimen can be considered as a sample from a very large population of volume elements in the base material. If all the elements in the sample are under equal stress, the fatigue life of the specimen will depend on the strength of the weakest element. The strength of the weakest element will vary due to sampling effects and thus give rise to the characteristic variability in observed fatigue life. The same reasoning applies when the stress is not uniform throughout the specimen, but the situation is then more complex. It is interesting to note that this point of view explains the observation that large specimens tend to be weaker than small specimens. This follows from the fact that the weakest element in a large sample of elements is likely to be weaker than the weakest element in a small sample. The smallest or the largest value in a sample is called an extreme value, and the behavior of such values is dealt with by what is called the theory of extreme values (**3**). The extreme value distribution discussed previously as the distribution of fatigue life at a fixed stress, follows directly from this theory.

The fundamental theories of fatigue behavior which involve probability and statistics should be distinguished from the methods discussed above for describing and analyzing observed data. A brief review and references to the various theories of fatigue failure are given by Stulen (**10**).

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A STUDY OF STATISTICAL TREATMENTS OF FATIGUE DATA*

BY M. N. TORREY[1] AND G. R. GOHN[1]

SYNOPSIS

This paper summarizes the results of reversed-bending fatigue tests on two lots of commercial Grade A (5 per cent tin) phosphor bronze strip and presents the observed values (48 values for each deflection level) so that others may use them for further statistical analysis. The distributions of cycle life are fitted by logarithmic-normal, extreme value and log-log-normal distributions, showing that the type of distribution depends on the deflection (stress) value. The use of response curves is suggested.

While the phenomenon of fatigue has been recognized for well over a century, the mechanism of fatigue failure has been studied intensively in this country only since 1919. At that time the National Research Foundation, in cooperation with the Engineering Foundation and the University of Illinois, undertook a cooperative study of fatigue. These studies were summarized in a manual on fatigue by H. F. Moore (**1**)[2] in 1927. Since that time our knowledge of the behavior of materials and mechanical designs subjected to repeated applications of stress or strain has increased markedly, but our understanding of the mechanism of fatigue failure is not yet complete.

New research tools such as the electron microscope have further increased our knowledge of the phenomenon while developments in solid state physics and in statistics have contributed much towards a more rational explanation of the causes of fatigue. Statistical methods, in particular, have been increasingly applied to the analysis of fatigue data in recent years. In 1951 and 1952, ASTM Committee E-9 on Fatigue sponsored two symposia (**2**, **3**) on the statistical aspects of fatigue. Since that time a number of individually sponsored papers have appeared in the literature. Two of these papers, one by Schuette (**4**), the other by Gohn, Guerard, and Freynik (**5**) are of particular interest in that they discuss the statistical limitations of the usual type of fatigue test in which four or five specimens are tested at each of several different stress levels. A few others such as Sinclair and Dolan (**6**), Corten and Sinclair (**7**), and Ransom and Mehl (**8**), have made statistical studies on a larger number of specimens tested at the same stress level. So significant are these studies that Committee E-9 is now preparing a manual on the statistical treatment of fatigue data. However, the real need at present appears to be for sufficient data at a series of stress levels to permit application of various statistical treatments for their evaluation. In the present paper, the authors have made a preliminary analysis of observations on more than 1000 specimens tested in groups of 48 at 9 to 12 different deflection (stress) levels and have presented the original test data so that others interested in the statistical aspects of fatigue may work with these data.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1116.

MATERIALS INVESTIGATED

The materials used in these studies consisted of two lots of commercial grade A (5 per cent tin) phosphor bronze strip rolled (after an 0.035-mm ready-to-finish grain size anneal) from an ingot containing 0.05 per cent phosphorus and 5 per cent tin. Fifty feet of each material was furnished in flat lengths approximately 6 ft long, 8 in. wide, and 0.040 in. thick. One lot met the requirements for hard temper strip as specified in the ASTM Standard Specification for Phosphor Bronze Plate, Sheet, Strip and Rolled Bar (B 103 - 55);[3] the other met the requirements for spring-temper strip. These two materials had physical properties substantially the same as those shown in Fig. 13 of the paper, "The Mechanical Properties of Wrought Phosphor Bronze Alloys," by Gohn, Guerard, and Freynik (5) for the hard and spring tempers, 5 per cent tin bronze, that is, material cold rolled 37.4 and 60.5 per cent, respectively.

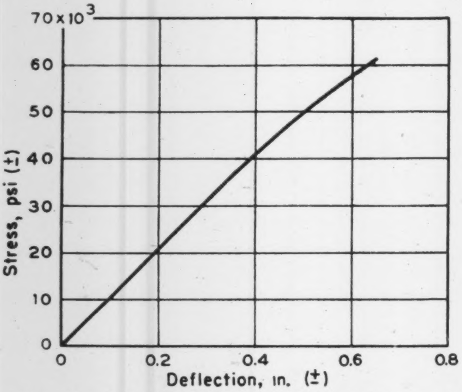

FIG. 1.—Relationship Between Deflection and Stress.

TEST SPECIMENS

In preparing the fatigue test specimens, the outer ½ in. of each 6-ft length of phosphor bronze strip was discarded. The remainder of the strip was cut into 11 lengths of approximately 6½ in. From each of these lengths, fourteen ½ by 6½ in. blanks were sheared parallel to the direction of rolling. After these blanks were well mixed, groups of 12 to 15 were drawn from the blanks, clamped together and machined by cross-milling with a spiral-fluted, contour cutter to produce fatigue test specimens which conformed to the dimensions shown in Fig. 10(*c*) of the Manual on Fatigue Testing (9). The maximum variation of the width of these specimens in the trapezoidal area of uniform stress did not exceed ±0.0005 in. from the nominal dimensions shown; this is less than ±0.26 per cent of the smallest critical dimension.

FATIGUE TESTS

The fatigue tests were all made on the same 24-specimen sheet metal fatigue machine designed by Gohn and Morton (10). Two runs of 24 specimens each were made for both tempers. Since a study of the distribution of cycle life at a given deflection level was the main purpose of this experiment, all four runs were made before changing the deflection of the fatigue testing machine. This deflection was measured to the nearest ±0.0001 in., and deviations between the deflection of the front arm (specimens 1 to 12) and the back arm (specimens 13 to 24) did not exceed 0.0015 in. Cycle life was recorded for each position to the nearest 1000 cycles; complete failure being taken as the end point. All tests were made at a temperature of 80 to 85 F and a speed of approximately 2000 cpm.

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[3] 1955 Book of ASTM Standards, Part 2, p. 160.

To permit conversion from deflection levels to stress levels, which have a more general application in fatigue studies, a special experiment was run to establish the relationship between deflection and stress for these materials. This relationship, which is shown in Fig. 1, was established by measuring the bending moment required to produce the given deflection at six different levels using ten separate test specimens whose average thickness was equal to the average of the lot.

THE DATA

Table I presents the observed values of cycle life-to-failure, up to 10^8, at zero mean stress for the hard temper strip. The data are given by positions for twelve deflection levels and for each run separately. The date of the beginning of each test is noted.

Several preliminary tests were run on other machines before data showing no significant position effect were obtained. Tests for randomness for all levels without runouts indicate that the data presented here are free from significant position effect. Figure 2 shows the data for three deflection levels. The arrangement of the observed values in the order of their positions on the fatigue machine is, from a statistical standpoint, considered to provide a random arrangement of their magnitudes.

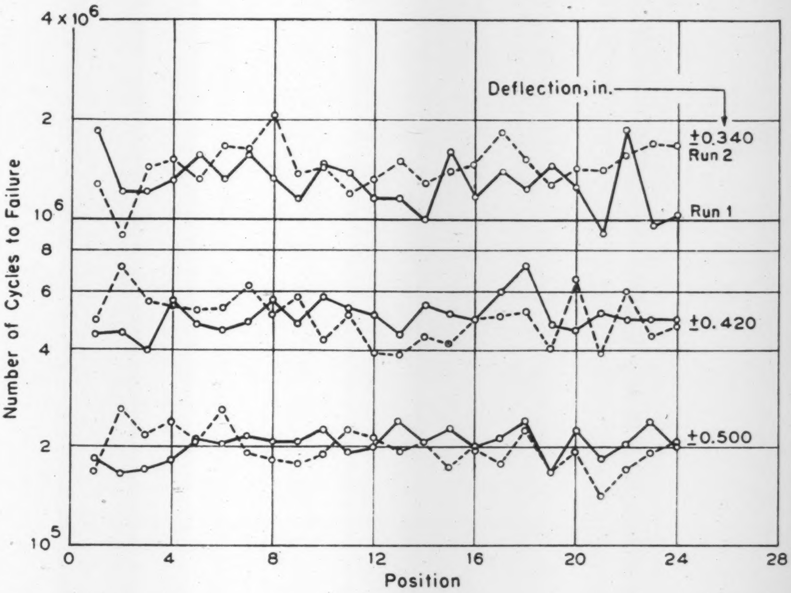

FIG. 2.—Number of Cycles-to-Failure by Runs and Position of Specimens on the Machine, Hard Temper, Grade A Phosphor Bronze Strip.

Table II presents comparable data for spring temper strip for nine deflection levels.

Tables III and IV present the same data rearranged so that the values are listed in the order of magnitude of cycle life, combining runs 1 and 2. The run 2 values are marked with asterisks. This arrangement facilitates the use of a "run test" (11, 12) to determine whether the data for runs 1 and 2 can be con-

TABLE I.—FATIGUE DATA FROM BENDING TESTS ON HARD TEMPER GRADE
GRAIN SIZE, 0.05 PER

Cycle Life to Failure at Zero

Test Deflection→	±0.540 In.	±0.500 In.	±0.460 In.	±0.420 In.	±0.380 In.
Date →	10/22/52	12/4/52	12/8/52	12/12/52	1/15/53
Position ↓					
1	118	184	423	444	688
2	129	167	484	454	732
3	141	171	426	394	624
4	135	181	438	556	781
5	148	213	383	476	699
6	105	204	296	458	774
7	153	215	297	483	865
8	131	208	327	563	662
9	120	208	340	475	948
10	119	226	393	575	681
11	148	192	357	531	807
12	128	200	348	503	724
13	134	240	397	441	906
14	134	209	396	542	610
15	117	228	400	505	659
16	124	196	337	487	741
17	125	216	305	599	665
18	107	239	330	715	696
19	108	166	377	473	585
20	126	225	326	456	689
21	122	185	367	509	584
22	103	205	314	490	703
23	114	237	283	489	679
24	89	200	410	487	938
Date →	10/23/52	12/5/52	12/9/52	12/15/52	1/16/53
Position ↓					
1	109	167	328	495	822
2	104	259	356	708	841
3	124	219	462	559	869
4	126	240	358	546	842
5	113	206	371	522	844
6	125	259	341	529	849
7	122	192	396	625	1,092
8	122	185	419	506	1,067
9	118	178	326	575	811
10	117	189	290	421	715
11	128	226	399	509	903
12	106	212	356	387	1,032
13	102	194	243	381	667
14	121	206	397	433	944
15	127	172	293	409	892
16	124	201	439	489	833
17	136	178	455	499	946
18	144	226	446	516	776
19	104	166	313	401	875
20	122	191	274	645	873
21	126	142	299	377	681
22	121	170	319	598	844
23	100	192	279	436	653
24	137	205	312	465	981

A (5 PER CENT TIN) PHOSPHOR BRONZE STRIP 0.035 MM READY-TO-FINISH CENT PHOSPHORUS.

Mean Stress (thousands)

±0.340 In.	±0.300 In.	±0.280 In.	±0.270 In.	±0.260 In.	±0.250 In.	±0.240 In.
Run 1						
2/10/53	2/27/53	7/17/53	1/25/54	9/3/53	4/7/54	6/23/54
1,845	2,994	13,558	55,331	100,000→	100,000→	100,000→
1,202	5,810	5,911	48,315	5,305	79,870	100,000→
1,205	3,547	10,020	69,212	49,734	100,000→	100,000→
1,300	5,568	4,578	15,393	44,476	100,000→	100,000→
1,549	2,717	3,378	6,151	31,431	100,000→	100,000→
1,302	25,049	3,850	41,983	100,000→	100,000→	100,000→
1,568	3,295	5,942	100,000→	83,576	100,000→	100,000→
1,333	55,525	4,174	100,000→	100,000→	100,000→	71,996
1,143	3,034	2,371	17,254	100,000→	100,000→	99,552
1,439	2,954	10,378	100,000→	100,000→	100,000→	90,498
1,368	14,255	8,375	4,431	77,665	100,000→	100,000→
1,136	7,250	10,487	97,948	100,000→	100,000→	100,000→
1,140	2,466	8,326	83,701	100,000→	78,836	47,358
988	3,504	14,293	14,369	5,975	38,896	100,000→
1,592	13,542	10,796	61,921	100,000→	100,000-	100,000→
1,163	2,999	5,487	15,842	100,000→	100,000→	100,000→
1,380	17,796	9,766	100,000→	100,000→	68,630	100,000→
1,214	6,401	1,713	73,357	9,519	86,388	100,000→
1,425	3,382	6,869	68,346	100,000→	100,000→	100,000→
1,235	2,660	6,228	63,043	10,750	100,000→	100,000→
899	17,912	2,466	91,965	100,000→	100,000→	100,000→
1,863	4,123	7,072	55,012	100,000→	100,000→	100,000→
947	3,382	3,981	93,851	100,000→	46,270	100,000→
1,019	3,361	3,553	100,000→	100,000→	100,000→	100,000→
Run 2						
2/16/53	3/9/53	7/28/53	3/2/54	10/8 /53	5/13/54	8/1 /54
1,287	2,464	7,076	37,983	100,000→	72,362	100,000→
898	2,534	4,202	56,871	57,727	76,399	100,000→
1,428	1,800	9,434	100,000→	100,000→	17,599	100,000→
1,507	18,040	3,787	90,026	60,338	100,000→	100,000→
1,303	50,716	5,965	100,000→	100,000→	100,000→	100,000→
1,663	2,404	3,776	82,568	100,000→	100,000→	100,000→
1,611	4,677	6,505	100,000→	33,794	100,000→	100,000→
2,042	76,074	10,133	83,079	100,000→	100,000→	100,000→
1,369	62,892	4,027	100,000→	87,905	100,000→	100,000→
1,423	12,678	6,180	77,548	100,000→	100,000→	66,340
1,189	1,688	71,283	100,000→	100,000→	23,978	53,301
1,299	1,796	74,758	100,000→	42,470	100,000→	100,000→
1,491	17,950	2,441	37,969	100,000→	100,000→	100,000→
1,285	73,501	100,000→	87,545	100,000→	8,204	100,000→
1,385	3,014	7,877	69,744	100,000→	100,000→	100,000→
1,460	50,432	9,356	37,957	100,000→	100,000→	100,000→
1,822	11,212	41,832	100,000→	100,000→	100,000→	100,000→
1,505	7,105	6,839	100,000→	100,000→	100,000→	100,000→
1,260	11,924	21,839	6,695	100,000→	99,002	100,000→
1,421	36,921	6,806	39,235	100,000→	95,608	100,000→
1,395	32,522	56,998	100,000→	100,000→	100,000→	100,000→
1,547	7,104	9,218	100,000→	100,000→	100,000→	100,000→
1,678	3,456	3,721	15,580	71,886	100,000→	100,000→
1,641	20,634	15,757	56,502	100,000→	100,000→	100,000→

TABLE II.—FATIGUE DATA FROM BENDING TESTS ON SPRING TEMPER GRADE
GRAIN SIZE, 0.05 PER

Cycle Life to Failure at Zero

Test Deflection→	±0.540 In.	±0.500 In.	±0.460 In.	±0.420 In.
Date →	10/24/52	12/3/52	12/10/52	12/16/52
Position ↓				
1	152	253	420	807
2	172	201	403	733
3	155	263	531	646
4	172	271	402	640
5	171	287	421	596
6	156	253	383	781
7	214	311	440	760
8	194	332	435	599
9	162	390	573	661
10	210	371	422	774
11	164	284	410	662
12	180	264	424	651
13	188	238	528	744
14	171	271	435	675
15	192	226	453	539
16	186	248	413	638
17	201	262	402	889
18	185	259	450	628
19	152	224	506	684
20	191	333	514	530
21	140	281	422	607
22	152	269	428	757
23	139	232	365	719
24	164	279	448	568
Date →	10/24/52	12/4/52	12/11/52	1/2/53
Position ↓				
1	156	275	430	477
2	164	244	345	567
3	189	240	415	628
4	163	245	412	646
5	166	281	400	772
6	161	252	430	648
7	195	300	483	821
8	186	309	422	592
9	155	273	421	546
10	188	292	446	747
11	156	257	409	556
12	189	279	399	494
13	156	282	415	665
14	150	258	458	672
15	161	252	395	639
16	176	279	474	685
17	164	296	465	493
18	195	254	436	655
19	181	230	405	527
20	164	277	416	590
21	152	251	412	621
22	176	278	387	858
23	182	285	409	594
24	176	307	592	778

A (5 PER CENT TIN) PHOSPHOR BRONZE STRIP 0.035 MM READY-TO-FINISH
CENT PHOSPHORUS.

Mean Stress (thousands)

	±0.380 In.	±0.360 In.	±0.340 In.	±0.300 In.	±0.260 In.
Run 1					
	1/5/53	7/28/55	1/21/53	4/16/53	11/13/53
	1,244	1,172	1,723	100,000→	100,000→
	1,023	1,050	12,950	100,000→	100,000→
	920	1,426	5,109	100,000→	100,000→
	819	1,597	1,440	100,000→	100,000→
	1,046	2,547	60,159	100,000→	100,000→
	1,041	1,643	17,983	100,000→	100,000→
	1,192	1,577	10,606	100,000→	100,000→
	1,011	2,037	72,855	100,000→	100,000→
	902	1,141	33,061	74,593	100,000→
	1,087	3,867	35,999	100,000→	100,000→
	867	1,179	25,031	100,000→	100,000→
	729	1,322	42,628	100,000→	100,000→
	827	1,399	22,771	100,000→	100,000→
	863	1,961	16,201	100,000→	100,000→
	868	1,378	42,919	100,000→	100,000→
	834	1,148	23,565	49,545	100,000→
	1,241	2,337	2,706	72,528	100,000→
	1,061	1,034	1,605	100,000→	100,000→
	889	1,123	4,350	100,000→	100,000→
	936	1,304	32,790	100,000→	100,000→
	715	1,562	9,503	100,000→	100,000→
	814	1,107	1,902	88,369	100,000→
	855	1,606	21,756	100,000→	100,000→
	1,162	986	19,705	100,000→	100,000→
Run 2					
	1/6/53	8/2/55	2/26/53	5/20/53	12/17/53
	1,311	7,432	1,419	53,602	100,000→
	990	2,025	2,450	100,000→	100,000→
	1,090	2,736	2,450	59,190	100,000→
	899	1,302	2,286	100,000→	100,000→
	1,464	1,909	2,913	100,000→	100,000→
	837	2,753	1,385	100,000→	100,000→
	1,021	8,400	49,038	100,000→	100,000→
	969	9,604	1,946	100,000→	100,000→
	928	1,909	1,653	72,339	100,000→
	1,217	1,970	4,070	13,368	100,000→
	1,200	1,778	1,989	100,000→	100,000→
	886	1,920	1,837	100,000→	100,000→
	1,185	10,291	20,462	100,000→	100,000→
	1,048	6,472	4,678	77,519	100,000→
	1,216	3,417	20,479	100,000→	100,000→
	1,009	1,422	1,855	100,000→	100,000→
	839	1,745	75,075	100,000→	100,000→
	1,310	4,306	5,676	83,463	100,000→
	1,038	2,620	1,557	100,000→	100,000→
	1,149	1,748	10,730	100,000→	100,000→
	887	1,797	14,228	100,000→	100,000→
	987	2,979	2,526	100,000→	100,000→
	849	3,194	1,308	100,000→	100,000→
	1,186	7,993	2,461	100,000→	100,000→

TABLE III.—FATIGUE DATA FROM BENDING TESTS ON HARD TEMPER GRADE
GRAIN SIZE, 0.05 PER

Cycle Life to Failure at Zero

Test Deflection→ Rank ↓	±0.540 In.	±0.500 In.	±0.460 In.	±0.420 In.	±0.380 In.
1	89	142*	243*	377*	584
2	100*	166	274*	381*	585
3	102*	166*	279*	387*	610
4	103	167	283	394	624
5	104*	167*	290*	401*	653*
6	104*	170*	293*	409*	659
7	105	171	296	421*	662
8	106*	172*	297	433*	665
9	107	178*	299*	436*	667*
10	108	178*	305	441	679
11	109*	181	312*	444	681
12	113*	184	313*	454	681*
13	114	185	314	456	688
14	117	185*	319*	458	689
15	117*	189*	326	465*	696
16	118	191*	326*	473	699
17	118*	192	327	475	703
18	119	192*	328*	476	715*
19	120	192*	330	483	724
20	121*	194*	337	487	732
21	121*	196	340	487	741
22	122	200	341*	489	774
23	122*	200	348	489*	776*
24	122*	201*	356*	490	781
25	122*	204	356*	495*	807
26	124	205	357	499*	811*
27	124*	205*	358*	503	822*
28	124*	206*	367	505	833*
29	125	206*	371*	506*	841*
30	125*	208	377	509*	842*
31	126	208	383	509	844*
32	126*	209	393	516*	844*
33	126*	212*	396	522*	849*
34	127*	213	396*	529*	865
35	128	215	397	531	869*
36	128*	216	397*	542	873*
37	129	219*	399*	546*	875*
38	131	225	400	556	892*
39	134	226	410	559*	903*
40	134	226*	419*	563	906
41	135	226*	423	575	938
42	136*	228	426	575*	944*
43	137*	237	438	598*	946*
44	141	239	439*	599	948
45	144*	240	446*	625*	981*
46	148	240*	455*	645*	1,032*
47	148	259*	462*	708*	1,067*
48	153	259*	484	715	1,092*

Notes:
* Second Run
→ No Failure

A (5 PER CENT TIN) PHOSPHOR BRONZE STRIP 0.035 MM READY-TO-FINISH CENT PHOSPHORUS.

Mean Stress (thousands)

±0.340 In.	±0.300 In.	±0.280 In.	±0.270 In.	±0.260 In.	±0.250 In.	±0.240 In.
898*	1,688*	1,713	4,431	5,305	8,204*	47,358
899	1,796*	2,371	6,151	5,975	17,599*	53,301*
947	1,800*	2,441*	6,695*	9,519	23,978*	66,340*
988	2,404*	2,466	14,369	10,750	38,896	71,996
1,019	2,464*	3,378	15,393	31,431	46,270	90,498
1,136	2,466	3,553	15,580*	33,794*	68,630	99,552
1,140	2,534*	3,721*	15,842	42,470*	72,362*	100,000
1,143	2,660	3,776*	17,254	44,476	76,399*	100,000→*
1,163	2,717	3,787*	37,957*	49,734	78,836	100,000→
1,189*	2,954	3,850	37,969*	57,727*	79,870	100,000→*
1,202	2,994	3,981	37,983*	60,338*	86,388	100,000→
1,205	2,999	4,027*	39,235*	71,886*	95,608*	100,000→*
1,214	3,014*	4,174	41,983	77,665	99,002*	100,000→
1,235	3,034	4,202*	48,315	83,576	100,000→	100,000→*
1,260*	3,295	4,578	55,012	87,905*	100,000→*	100,000→
1,285*	3,361	5,487	55,331	100,000→	100,000→	100,000→*
1,287*	3,382	5,911	56,502*	100,000→*	100,000→*	100,000→
1,299*	3,382	5,942	56,871*	100,000→	100,000→	100,000→*
1,300	3,456*	5,965*	61,921	100,000→*	100,000→*	100,000→
1,302	3,504	6,180*	63,043	100,000→	100,000→	100,000→*
1,303*	3,547	6,228	68,346	100,000→*	100,000→*	100,000→
1,333	4,123	6,505*	69,212	100,000→	100,000→	100,000→*
1,368	4,677*	6,806*	69,744*	100,000→*	100,000→*	100,000→
1,369*	5,568	6,839*	73,357	100,000→	100,000→	100,000→*
1,380	5,810	6,869	77,548*	100,000→*	100,000→*	100,000→
1,385*	6,401	7,072	82,568*	100,000→	100,000→	100,000→*
1,395*	7,104*	7,076*	83,079*	100,000→*	100,000→*	100,000→
1,421*	7,105*	7,877*	83,701	100,000→	100,000→	100,000→*
1,423*	7,250	8,326	87,545*	100,000→*	100,000→*	100,000→
1,425	11,212*	8,375	90,026*	100,000→	100,000→	100,000→*
1,428*	11,924*	9,218*	91,965	100,000→*	100,000→*	100,000→
1,439	12,678*	9,356*	93,851	100,000→	100,000→	100,000→*
1,460*	13,542	9,434*	97,948	100,000→*	100,000→*	100,000→
1,491*	14,255	9,766	100,000→	100,000→	100,000→	100,000→*
1,505*	17,796	10,020	100,000→*	100,000→*	100,000→*	100,000→
1,507*	17,912	10,133*	100,000→	100,000→	100,000→	100,000→*
1,547*	17,950*	10,378	100,000→*	100,000→*	100,000→*	100,000→
1,549	18,040*	10,487	100,000→	100,000→	100,000→	100,000→*
1,568	20,634*	10,796	100,000→*	100,000→*	100,000→*	100,000→
1,592	25,049	13,558	100,000→	100,000→	100,000→	100,000→*
1,611*	32,522*	14,293	100,000→*	100,000→*	100,000→*	100,000→
1,641*	36,921*	15,757*	100,000→	100,000→	100,000→	100,000→*
1,663*	50,432*	21,839*	100,000→*	100,000→*	100,000→*	100,000→
1,678*	50,716*	41,832*	100,000→*	100,000→	100,000→	100,000→*
1,822*	55,525	56,998*	100,000→*	100,000→*	100,000→*	100,000→
1,845	62,892*	71,283*	100,000→*	100,000→*	100,000→	100,000→*
1,863	73,501*	74,758*	100,000→*	100,000→*	100,000→*	100,000→*
2,042*	76,074*	100,000→*	100,000→*	100,000→*	100,000→	100,000→*

TABLE IV.—FATIGUE DATA FROM BENDING TESTS ON SPRING TEMPER GRADE
GRAIN SIZE, 0.05 PER

Cycle Life to Failure at Zero

Test Deflection→ Rank↓	± 0.540 In.	± 0.500 In.	± 0.460 In.	± 0.420 In.
1	139	201	345*	477*
2	140	224	365	493*
3	150*	226	383	494*
4	152	230*	387*	527*
5	152*	232	395*	530
6	152	238	399*	539
7	152	240*	400*	546*
8	155	244*	402	556*
9	155*	245*	402	567*
10	156	248	403	568
11	156*	251*	405*	590*
12	156*	252*	409*	592*
13	156*	252*	409*	594*
14	161*	253	410	596
15	161*	253	412*	599
16	162	254*	412*	607
17	163*	257*	413	621*
18	164	258*	415*	628
19	164*	259	415*	628*
20	164	262	416*	638
21	164*	263	420	639*
22	164*	264	421	640
23	166*	269	421*	646
24	171	271	422	646*
25	171	271	422*	648*
26	172	273*	422	651
27	172	275*	424	655*
28	176*	277*	428	661
29	176*	278*	430*	662
30	176*	279	430*	665*
31	180	279*	435	672*
32	181*	279*	435	675
33	182*	281	436*	684
34	185	281*	440	685*
35	186	282*	446*	719
36	186*	284	448	733
37	188	285*	450	744
38	188*	287	453	747*
39	189*	292*	458*	757
40	189*	296*	465*	760
41	191	300*	474*	772*
42	192	307*	483*	774
43	194	309*	506	778*
44	195*	311	514	781
45	195*	332	528	807
46	201	333	531	821*
47	210	371	573	858*
48	214	390	592*	889

Notes:
*Second Run
→No Failure

A (5 PER CENT TIN) PHOSPHOR BRONZE STRIP 0.035 MM READY-TO-FINISH
CENT PHOSPHORUS.

Mean Stress (thousands)

± 0.380 In.	± 0.360 In.	± 0.340 In.	± 0.300 In.	± 0.260 In.
715	986	1,308*	13,368*	100,000→
729	1,034	1,385*	49,545	100,000→*
814	1,050	1,419*	53,602*	100,000→
819	1,107	1,440	59,190*	100,000→*
827	1,123	1,557*	72,339*	100,000→
834	1,141	1,605	72,528	100,000→*
837*	1,148	1,653*	74,593	100,000→
839*	1,172	1,723	77,519*	100,000→*
849*	1,179	1,837*	83,463*	100,000→
855	1,302*	1,855*	88,369	100,000→*
863	1,304	1,902	100,000→	100,000→
867	1,322	1,946*	100,000→*	100,000→*
868	1,378	1,989*	100,000→	100,000→
886*	1,399	2,286*	100,000→*	100,000→*
887*	1,422*	2,450*	100,000→	100,000→
889	1,426	2,450*	100,000→*	100,000→*
899*	1,562	2,461*	100,000→	100,000→
902	1,577	2,526*	100,000→*	100,000→*
920	1,597	2,706	100,000→	100,000→
928*	1,606	2,913*	100,000→*	100,000→*
936	1,643	4,070*	100,000→	100,000→
969*	1,745*	4,350	100,000→*	100,000→*
987*	1,748*	4,678*	100,000→	100,000→
990*	1,778*	5,109	100,000→*	100,000→*
1,009*	1,797*	5,676*	100,000→	100,000→
1,011	1,909*	9,503	100,000→*	100,000→*
1,021*	1,909*	10,606	100,000→	100,000→
1,023	1,920*	10,730*	100,000→*	100,000→*
1,038*	1,961	12,950	100,000→	100,000→
1,041	1,970*	14,228*	100,000→*	100,000→*
1,046	2,025*	16,201	100,000→	100,000→
1,048*	2,037	17,983	100,000→*	100,000→*
1,061	2,337	19,705	100,000→	100,000→
1,087	2,547	20,462*	100,000→*	100,000→*
1,090*	2,620*	20,479*	100,000→	100,000→
1,149*	2,736*	21,756	100,000→*	100,000→*
1,162	2,753*	22,771	100,000→	100,000→
1,185*	2,979*	23,565	100,000→*	100,000→*
1,186*	3,194*	25,031	100,000→	100,000→
1,192	3,417*	32,790	100,000→*	100,000→*
1,200*	3,867	33,061	100,000→	100,000→
1,216*	4,306*	35,999	100,000→*	100,000→*
1,217*	6,472*	42,628	100,000→	100,000→
1,241	7,432*	42,919	100,000→*	100,000→*
1,244	7,993*	49,038*	100,000→	100,000→
1,310*	8,400*	60,159	100,000→*	100,000→*
1,311*	9,604*	72,855	100,000→	100,000→
1,464*	10,291*	75,075*	100,000→	100,000→*

sidered to be from the same universe or not.

To avoid confusion between the experimental runs and the "runs" having asterisks, it seems desirable to refer to number of "groups" rather than the number of "runs." To carry out the test, the number of groups of numbers with asterisks and numbers with no asterisks are counted. For example, for deflection level ±0.540 in. in Table III, there are 29 groups for the 48 observed values.

TABLE V.—NUMBER OF GROUPS.

Deflection, in.	Hard Temper		Spring Temper	
	Critical Value[a]	Observed	Critical Value[a]	Observed
±0.540.........	17	29	17	25
±0.500.........	17	23	17	19
±0.460.........	17	30	17	23
±0.420.........	17	22	17	28
±0.380.........	17	18	17	24
±0.360.........	...	...	17	12[b]
±0.340.........	17	19	17	25
±0.300.........	17	19	2	6
±0.280.........	17	20		
±0.270.........	11	15		
±0.260.........	4	6		
±0.250.........	3	5		
±0.240.........	2	3		

[a] This is the lower 2.5 per cent level and is used as follows: When there are 17 or more groups in the 48 observed values the two runs can be considered as two samples from the same universe. The critical number is smaller where there are runouts, as indicated.

[b] Significantly fewer than expected.

For two samples of 24, the expected number of groups is 25; for a significance level of 2.5 per cent, the critical value, $u_{.025}$, is 17 (13). If there are fewer than 17 groups in the 48 observed values for a deflection level, the hypothesis that the two samples are independent random samples from the same universe would be rejected with a risk of being wrong equal to 2.5 per cent. If there are 17 or more groups, the two samples would be treated as if they were from the same universe. This test is not as powerful as other tests which assume a particular distribution of the universe values, for example a normal distribution, but it has the advantage that it is valid for any universe having a continuous distribution provided the samples are drawn at random independently (11).

Table V gives the number of groups in the data of Tables III and IV. Whereas the critical value is 17 for two samples of 24, the critical values for the low deflection levels are smaller; there are fewer observed values since the run-outs are not counted. Therefore, the critical value corresponding to each deflection level and each temper is given.

Only one value of number of groups is significantly low—that for ±0.360 in., spring temper. All other values indicate that the two runs may be treated as one sample. However, further analysis showed that the data for three other deflection levels, ±0.380 in. and ±0.280 in. for hard temper and ±0.340 in. for spring temper, though they meet the "run test," are in difficulty, as mentioned later.[4]

OBSERVED FREQUENCY DISTRIBUTIONS

The main purpose of this experiment was to study the distributions of cycle life values at several deflection (stress) levels. Figures 3 to 6 present the observed distributions in the form of frequency histograms (14). These charts do not include data for the deflection levels for which there are run-outs, except for ±0.280 in., hard temper, for which there was only one run-out. The data for the lower deflection levels are shown in the cumulative distributions of Figs. 11 and 12.

Figures 3 and 4 show the frequency distributions for runs 1 and 2 separately. The distributions for spring temper tested at ±0.360 in. deflection, which were shown to be different by the "run test," appear different when plotted.

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[4] See pp. 1105 and 1109.

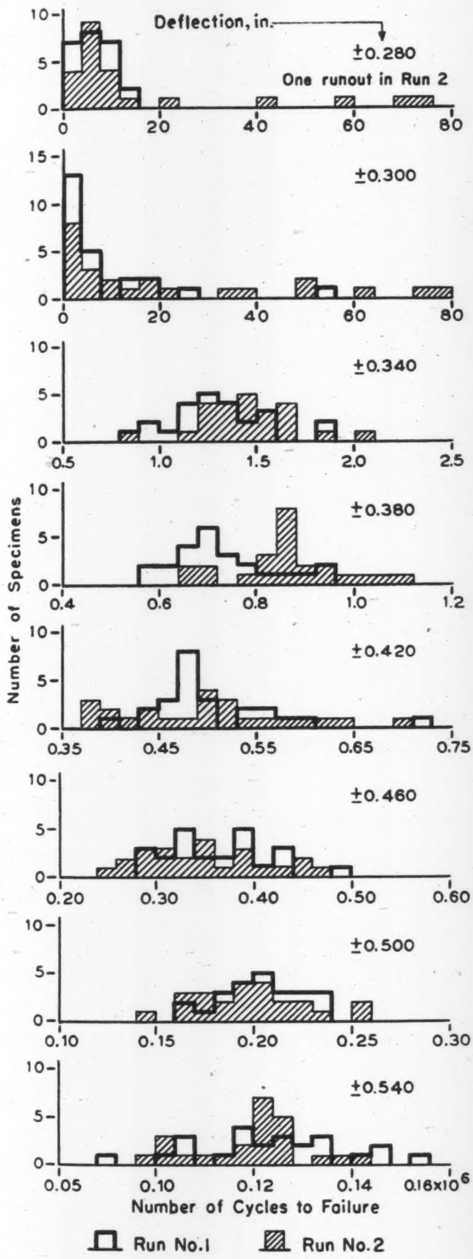

FIG. 3.—Frequency Histograms of Cycle Life, Runs 1 and 2 Separate, Hard Temper, Grade A Phosphor Bronze Strip.

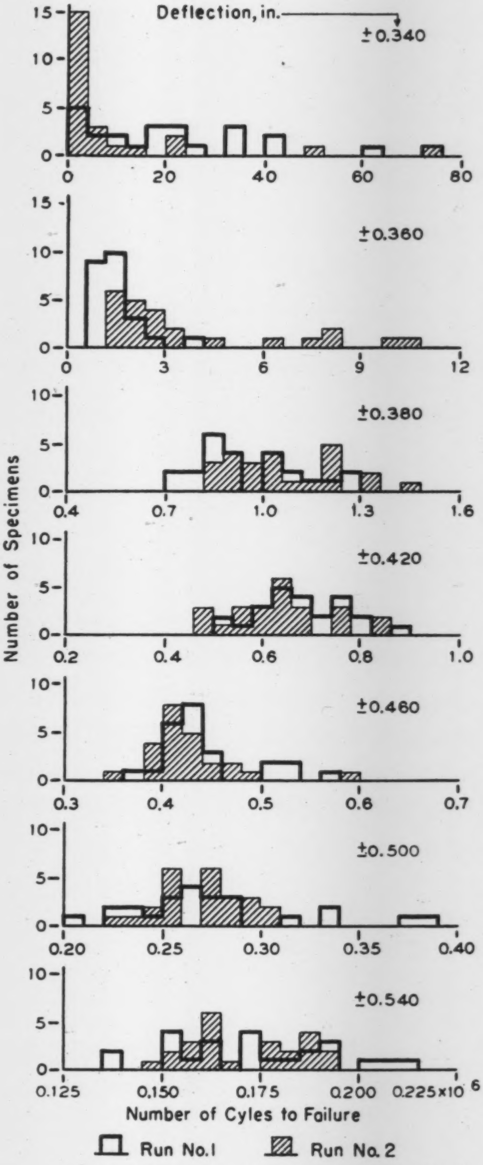

FIG. 4.—Frequency Histograms of Cycle Life, Runs 1 and 2 Separate, Spring Temper, Grade A Phosphor Bronze Strip.

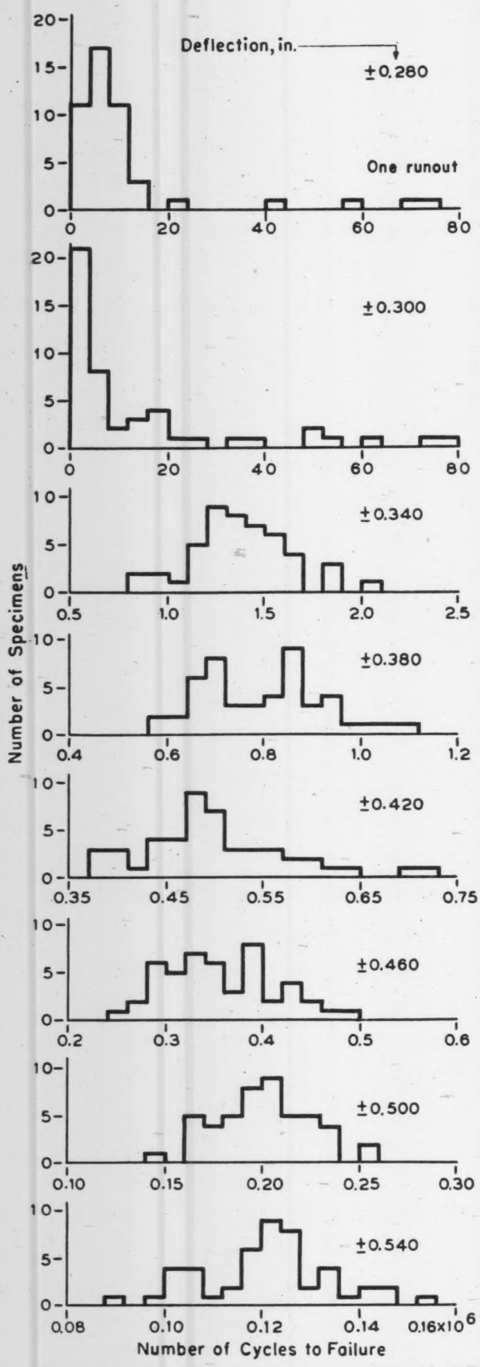

FIG. 5.—Frequency Histograms of Cycle Life, Runs 1 and 2 Combined, Hard Temper, Grade A Phosphor Bronze Strip.

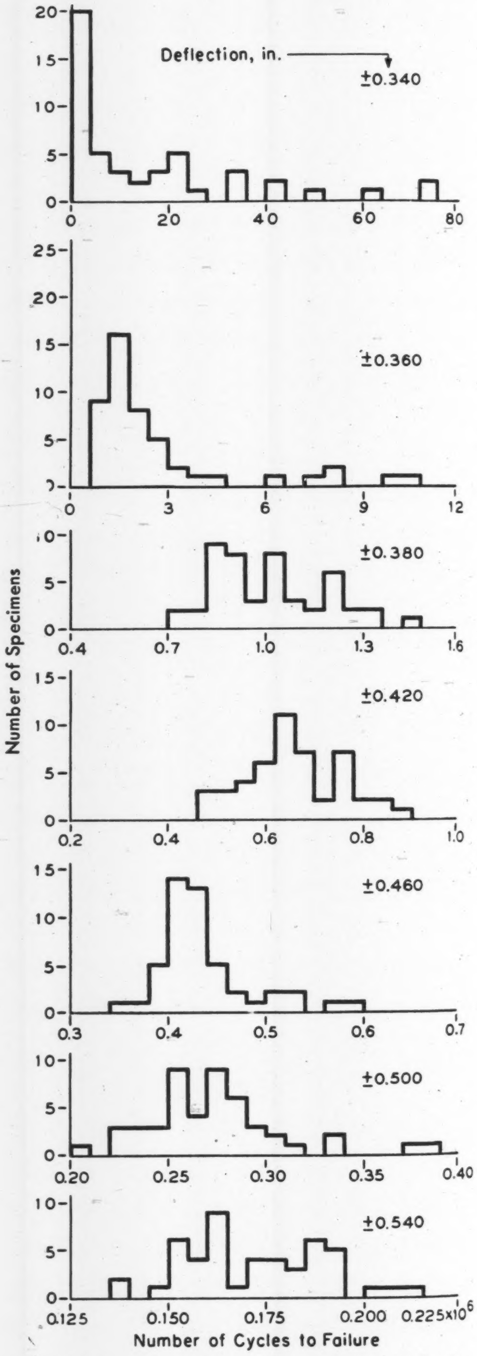

FIG. 6.—Frequency Histograms of Cycle Life, Runs 1 and 2 Combined, Spring Temper, Grade A Phosphor Bronze Strip.

Also, the distributions for hard temper tested at ±0.380 in. deflection look quite different; the number of observed groups, 18, is close to the critical value and thus the distributions are close to being called significantly different. Most of the pairs of distributions look like two random samples from one population.

As mentioned earlier, the four runs of 24 specimens (two runs for each of two tempers) were finished in each case before the deflection of the fatigue machine was changed. The fact that even two successive runs appear to be different raises a question of how much variability is introduced by the test when specimens are tested one at a time as is usual on many types of fatigue testing machines.

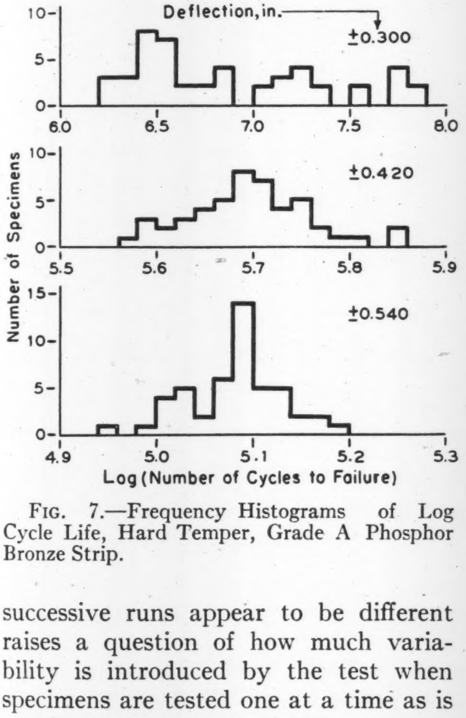

FIG. 7.—Frequency Histograms of Log Cycle Life, Hard Temper, Grade A Phosphor Bronze Strip.

Figures 5 and 6 show the frequency distributions of cycle life obtained by combining the total data of the two runs to give a better picture of the various distribution shapes. Figures 7 to 14 show the combined data for the several deflection levels; the observed differences between runs will be taken into consideration in the interpretation of the results.

THEORETICAL DISTRIBUTIONS

Forty-eight observed values are not enough to determine conclusively what theoretical distribution best fits the data. However, the frequency histograms in Figs. 5 and 6 indicate that the universe distributions may be skewed, especially for the small deflection values, and that the skewness, if any, is positive. Thus it is natural to try fitting the data with a log-normal distribution.

Figure 7 shows frequency histograms for three deflection levels (hard temper); the abscissa values are the logarithms of cycle life. The histograms for the deflection levels ±0.540 in. and ±0.420 in. appear to be approximately symmetrical, but the histogram for ±0.300 in. is still decidedly skewed. Would the "extreme value" distribution be better?

There are two "ultimate" extreme value distributions, the distribution of minima, which corresponds to the "weakest link" theory (15), and the distribution of maxima. The distribution of minima is skewed to the left (negative skewness) while the distribution of maxima is skewed to the right (positive skewness). Thus the extreme value distribution for maxima would be appropriate for data which are skewed to the right as are these data.

Figure 8 shows the cumulative frequency distributions (14) for four deflection levels (hard temper) plotted on log-normal probability paper. The abscissa scale is the normal probability scale and the ordinate scale is logarithmic. The values of Table III were used in plotting the observed data.

The plotting position on the abscissa scale is $m/49$, where $m =$ rank as given in Table III. The use of $\frac{m}{n+1}$ as the plotting position, as well as the use of

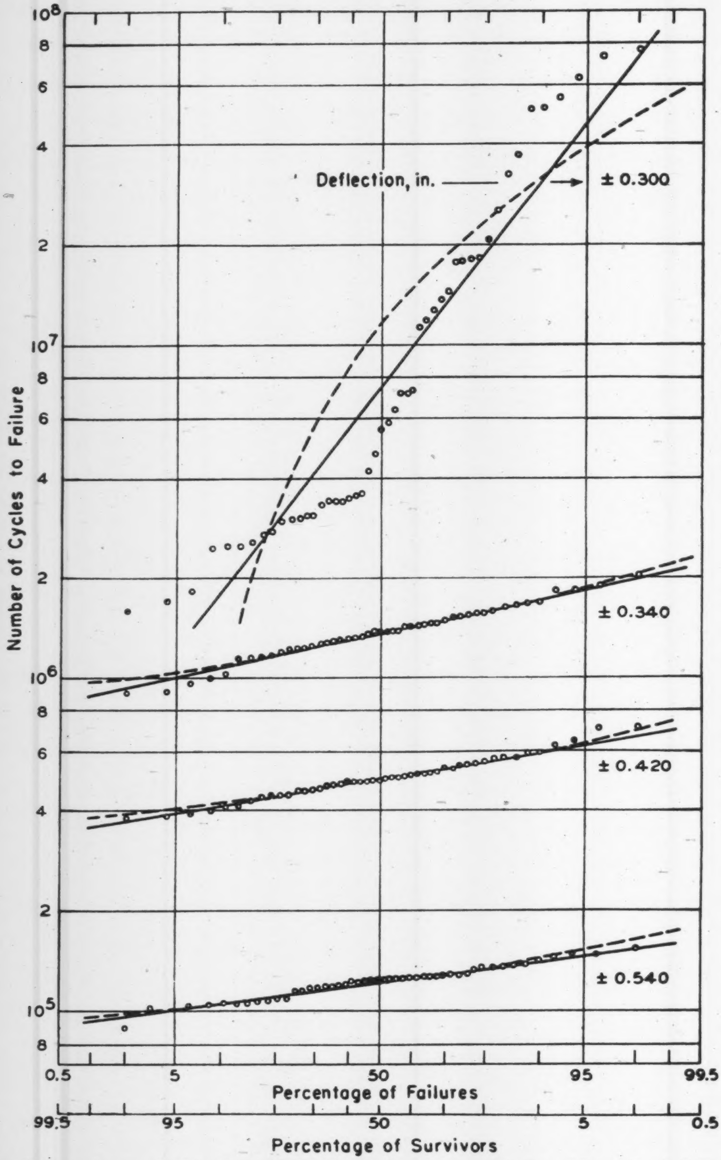

FIG. 8.—Comparison of the Cumulative Distributions with Log-Normal and Extreme Value Distributions, Hard Temper, Grade A Phosphor Bronze Strip.

the normal probability scale as the abscissa instead of the ordinate (contrary to usual custom), is recommended in a publication of the National Bureau of Standards (16). For this type of data, the values are observed in order of failure, and thus the rank may be considered to be the "fixed" variable.

The solid straight lines through the plotted points represent the theoretical log-normal distribution fitted[5] to the data by the use of the average and standard deviation values. The dashed curves are the extreme value distributions for maxima fitted[5] to the data by the method of order statistics (17). It has been pointed out (17) that this new method of fitting the extreme value distribution to observed data is more efficient than the methods proposed by Gumbel (16). The dashed curves would, of course, be straight lines on extreme value probability paper.

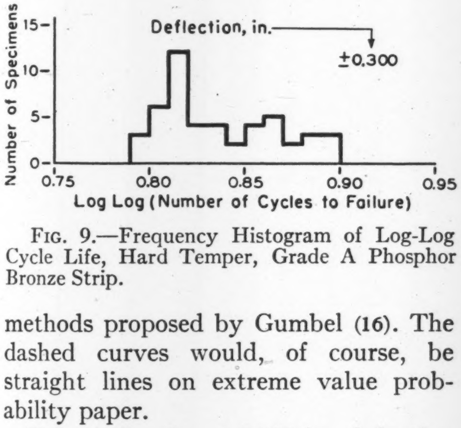

FIG. 9.—Frequency Histogram of Log-Log Cycle Life, Hard Temper, Grade A Phosphor Bronze Strip.

Except for the ±0.300-in. deflection level (Fig. 8), the log-normal straight lines and the extreme value curves differ only at the ends, corresponding to the tails of the distribution. It would take perhaps hundreds of observations at each stress level to show statistically which distribution is better. Even then it might not be possible since some of the observed values in the tails seem to come from another distribution—for example, the "early" failures for deflection level ±0.340 in.

Figure 8 shows quite dramatically the sudden change in distribution from the deflection level ±0.340 in. to the level ±0.300 in. The data for the low deflection levels have distributions quite different from those for the higher deflection levels for which the failures occur in a relatively short period of time. Neither the log-normal nor the extreme value distribution fits the data for the ±0.300 in. deflection.[6]

A frequency histogram of the observed data for the deflection level ±0.300 in. (hard temper) is shown in Fig. 9. The abscissa values are the logarithms of the logarithms of cycle life. Whereas this histogram is still not symmetrical, it looks more compact than the corresponding histogram in Fig. 7.

The cumulative distribution of these same data is shown in Fig. 10. The abscissa scale is the normal probability scale as in Fig. 8, but the ordinate scale, though labeled as cycles, is log-logarithmic (the logarithms of the logarithms of cycle life). The solid straight line represents the theoretical log-log-normal distribution fitted to the observed data. The dashed curve is the log-extreme-value distribution (the extreme value distribution fitted to the logarithms of the observed values). The dashed curve and straight line differ appreciably only in the left tail, where the log-extreme-value distribution appears to be better. The chi-square test for "goodness of fit" (18) gives no definite indication that one distribution is better than the other.

The conclusions from this study of fitting log-normal and extreme value distributions to these observed data are:

1. The log-normal distributions may

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[5] The equations of these fitted curves appear in the appendix.

[6] The computations of the extreme value curve has been thoroughly checked; the cause of the change in shape has not been determined.

be used for the finite life end[7] of the deflection-cycle life curve, that is, the higher deflection levels: ±0.340 in. and above for hard temper and ±0.380 in. and above for spring temper.

2. The log-log-normal distribution may be used for the infinite life end of the deflection-cycle life curve, that is, the lower deflection levels. The extreme value distribution does not appear to fit better.

Figures 11 and 12 show the cumulative distributions for most of the deflection levels[8] with a break in each ordinate scale to take care of the two different scales for the two ranges of deflection levels. The solid straight lines represent the log-normal or log-log-normal distributions, depending upon the range of deflection levels, which were computed from the data. For purposes of this preliminary investigation, the dashed lines were drawn parallel to the fitted log-log-normal lines, that is, parallel to the lines for ±0.300 in. deflection, hard temper, and ±0.360 in. deflection, spring temper. They all appear to fit the data except for the ±0.340 in. deflection level for spring temper; the reason for the excess of low values of number of cycles to failure is unknown. Most of the low values were in run 2 as shown in Fig. 4.

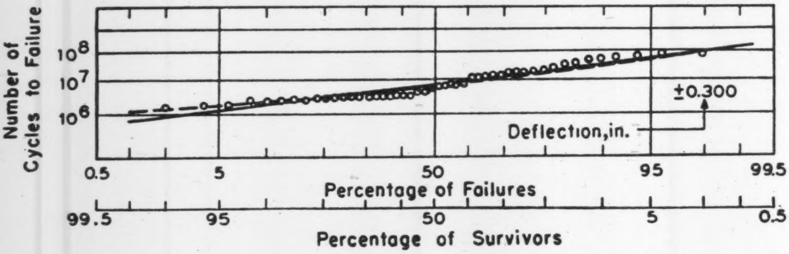

FIG. 10.—Comparison of a Cumulative Distribution with Log-Log-Normal and Log-Extreme-Value Curves, Hard Temper, Grade A Phosphor Bronze Strip.

DEFLECTION-CYCLE LIFE CURVES

The plotted points on the deflection-cycle life curves[9] of Fig. 13 (which can be converted to the conventional stress-cycle life, *S-N*, curves by use of the relationship for converting deflection to stress given in Fig. 1) are the cycle life values corresponding to the intersections of the fitted lines with the lines for percentage of failures equal to 5, 50, and 95 in Figs. 11 and 12. A definite break in the 5 per cent and 95 per cent curves appears between the deflection levels of ±0.340 in. and ±0.300 in. for hard temper, as well as between ±0.380 in. and ±0.360 in. for spring temper, because of the abrupt change in spread of the cycle life values. Beginning with the ±0.300 in. deflection level for hard temper and the ±0.360 in. deflection level for spring temper, the curves are broken because the points for the smaller deflection levels are based on the dashed lines of Figs. 11 and 12 which were not actually computed (as noted previously).

The 50 per cent curve corresponds to the median value of cycle life for each

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[7] The terms "finite life end" and "infinite life end" are used rather loosely to describe the parts of the deflection-cycle life curves before and after the "knee". (See Fig. 13).

[8] The curves for ±0.260 in. and ±0.280 in. deflections were omitted in Fig. 11 because they cross adjacent curves.

[9] The deflection scale should be the abscissa and the number of cycles the ordinate, from a statistical standpoint, but the method used is the conventional one for plotting this type of curve.

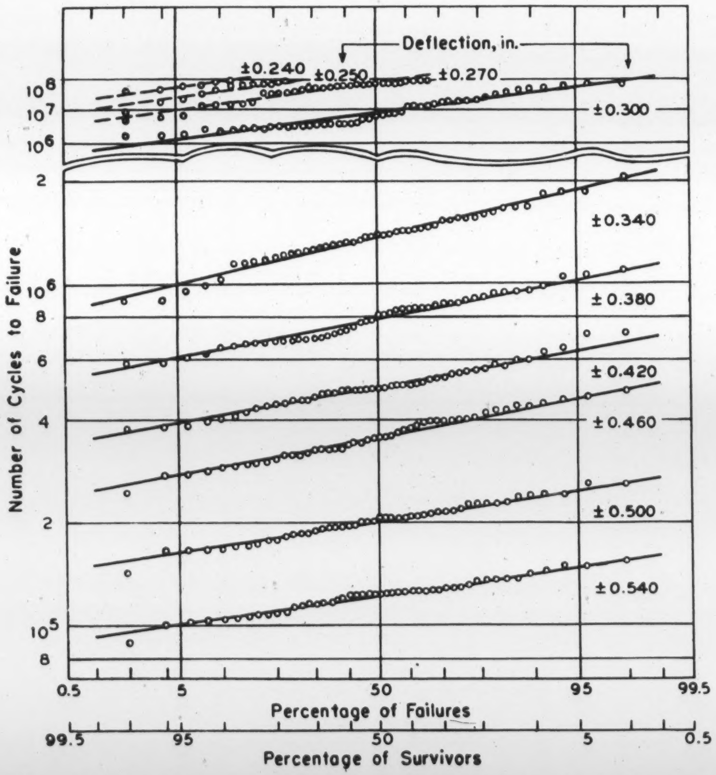

FIG. 11.—Cumulative Distributions, Hard Temper, Grade A Phosphor Bronze Strip.

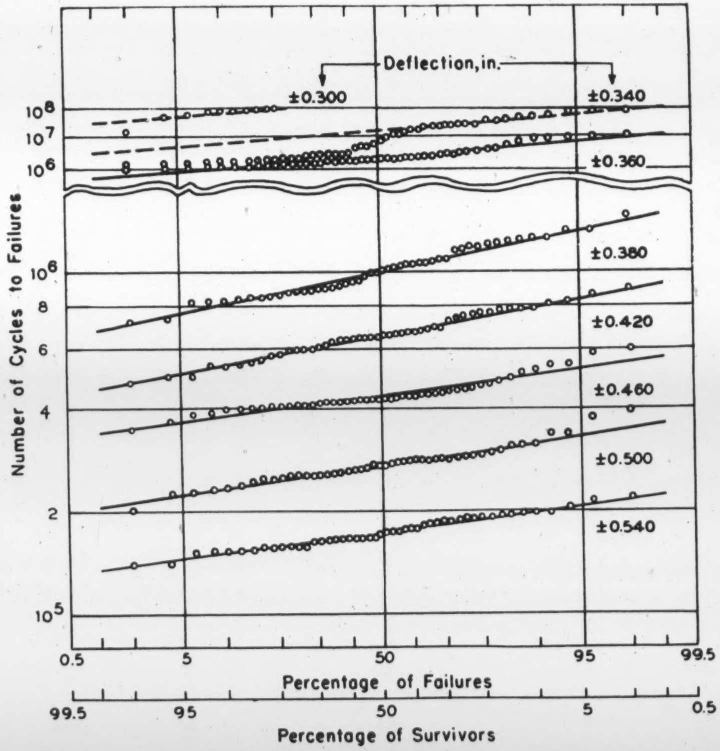

FIG. 12.—Cumulative Distributions, Spring Temper, Grade A Phosphor Bronze Strip.

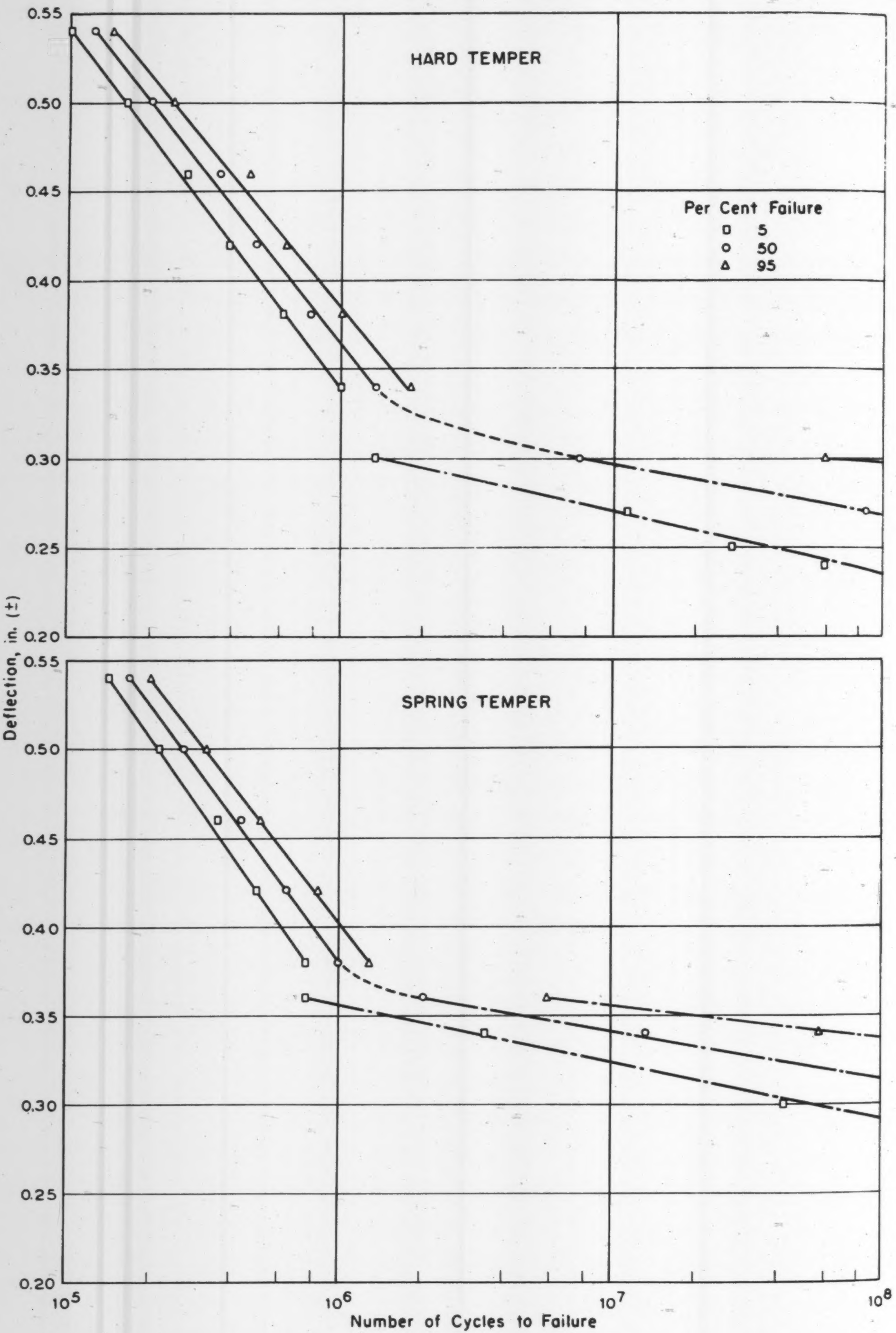

FIG. 13.—Deflection-Cycle Life Curves, Hard Temper and Spring Temper, Grade A Phosphor Bronze Strip.

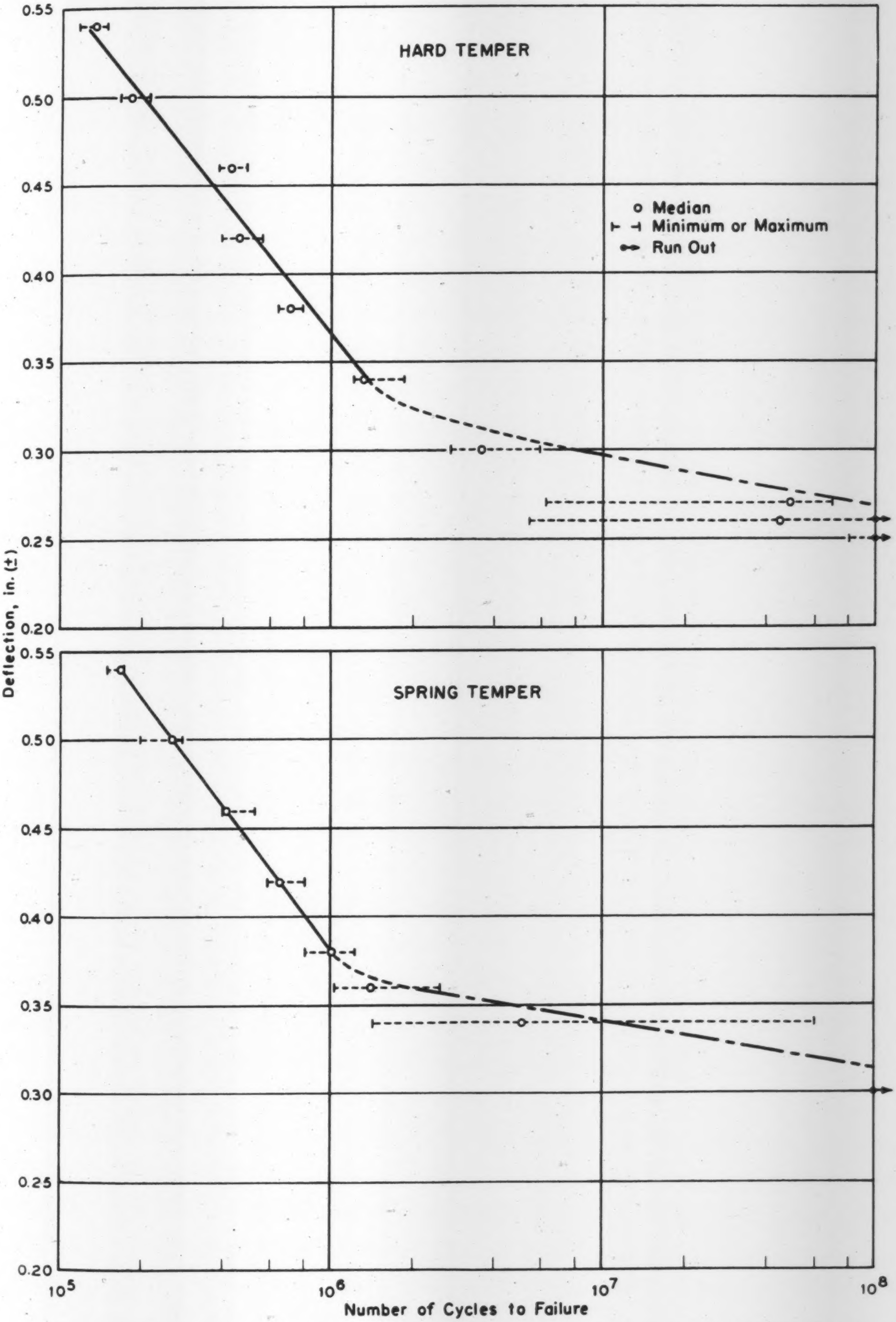

FIG. 14.—Comparison of the 50 Per Cent Curve (Fig. 13) and Medians of Samples of 5, Grade A Phosphor Bronze Strip.

deflection level. The 95 per cent and the 5 per cent curves correspond to the mean $\pm 1.65\sigma$ values, where σ is the standard deviation of individuals. These curves were computed in terms of the transformed values, logarithms for the finite life end and logarithms of logarithms for the infinite life end, and then converted to values of cycles-to-failure.

The curves shown in Fig. 13 are based on 48 observed values at each of the several deflection levels. Seldom, however, are 48 specimens tested; sometimes only one specimen is available for test, more frequently 4 or 5 specimens are tested at each deflection (stress) level. Hence a comparison of the results of tests made on 48 specimens with those made on only 5 specimens (the number normally used at the Bell Telephone Laboratories) has seemed desirable. Figure 14 shows a comparison between the 50 per cent curve, based upon 48 values, and the medians of samples of 5 (small circles). The minima and the maxima of the samples of 5 are included on the same chart (as the ends of the dashed lines). These samples are the first 5 values of run 1, corresponding to positions 1 to 5 as given in Tables I and II. Since the arrangement of the observed values by positions can be treated as a random arrangement of their magnitudes,[10] this may be considered to be a random sample of 5 from the larger sample.

At the finite life end of the curves, the 50 per cent curve fits the medians of 5 specimens fairly well for hard temper and phenomenally well for spring temper. This is not the case for the infinite life end of the curve; many more specimens are needed for estimating median life with the same precision—that is, the same width of confidence limits—in that region due to the large increase in variability. When the purpose of the test is to estimate median life at several deflection (stress) levels, it probably would be better to test more specimens in the region of infinite life than in the region of finite life, instead of testing the same number of specimens at each deflection (stress) level. This is of particular importance when only a few specimens are available for test. The transition region warrants further study; for example, several specimens tested at the ±0.320-in. deflection level for hard temper would provide additional information about the shapes of the curves as well as about the distributions.

An advantage of testing a larger number of specimens in regions of greater interest is the improvement in the prediction that can be made about the percentage of universe values below the sample minimum. For samples of 5 it may be said that in 90 per cent of the samples the percentage below the observed minimum will not exceed 37 per cent (**19**). For samples of 20, the percentage would not exceed 11 per cent in 90 per cent of the samples; for samples of 48 the percentage would not exceed 4.7 per cent in 90 per cent of the samples. These statements are based on nonparametric statistics and assume no particular distribution of cycle life values. Even though a theoretical distribution has been fitted to values of cycle life, it is safer to make statements about the tails of the distribution in terms of nonparametric statistics, since the theoretical distributions are only known, experimentally, to fit in the middle, and since not all theoretical distributions which appear to fit in the middle give the same results in the tails, as shown in Figs. 11 and 12.

RESPONSE CURVES

From the intersections of the fitted lines and a constant value of cycle life, such as 10^6, 10^7, or 10^8, the expected percentage of failures for a given deflection level may be read from Figs. 11 and

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[10] See p. 1093.

12. The deflection levels used are not really close enough together to plot "response" or "probit" curves (20) for the percentage of specimens having cycle life values less than a particular value. If the experiment had been designed for that type of analysis, the deflection levels used would have been grouped so that several fitted lines would intersect the lines corresponding to the particular values of cycle life for which curves are wanted.

An example of this type of chart is shown in Fig. 15. The values of percentage of failures before 10^6, 10^7, and 10^8 cycles were plotted on the normal probability scale because it tends to straighten response curves of this sort into straight lines. For this chart, the deflection values (not the percentages) are used for the abscissa scale because they are the "fixed" values. The values of deflection were chosen in discrete steps and are the independent variable, whereas the percentage of failures may have any value from 0 to 100 and is the dependent variable. The lines connecting the observed points are dashed because there are too few points for determining the lines accurately.

Since lines like those in Fig. 15, which estimate the means of binomial distributions, do not change drastically from one range of deflection levels to another, it is possible that this type of analysis might replace analyses requiring the fitting of cycle life distributions. The response curves are already being used (21) for locating the endurance limit, the deflection level (stress) corresponding to 50 per cent failures before 10^8 (or some other number) of cycles. They may be used for other percentage points and other values of number of cycles if the experiment is designed so as to give several values for each curve. In that case, the response curves would be fitted to the observed values of p, the per-

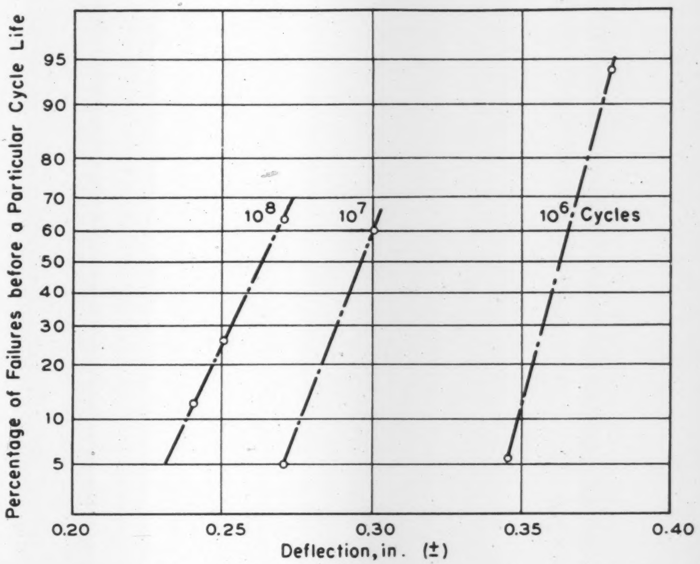

Fig. 15.—Response Curves Showing the Relation Between Percentage of Failures, Deflection and Cycle Life, Hard Temper, Grade A Phosphor Bronze Strip.

centage of failures, directly without fitting the distributions of cycle life values.

CONCLUSIONS

On the basis of these studies the authors conclude that:

1. For the theoretical distributions investigated, the type of distribution of cycle life values at the infinite life end of the deflection-cycle life or S-N curve is different from the type of distribution at the finite life end.

2. In designing an experiment for the purpose of making a deflection-cycle life or an S-N diagram for materials like those tested, the number of specimens per deflection level tested at the infinite life end of the curve should be considerably larger than the number tested at the finite life end.

3. Since the change in the type of distribution for small *versus* large deflection levels does not seem to have an appreciable effect on response curves, their use for several values of cycle life, such as 10^6, 10^7, and 10^8, should be investigated further.

Acknowledgment:

The authors wish to express their appreciation to J. P. Ahrens and A. Fox who assisted in the fatigue tests, to Miss J. Zagrodnick for making computations and to the Misses Elsa Kerner and A. G. Loe for preparing the illustrations.

APPENDIX I

The equations of the fitted lines and curves shown in Figs. 8, 11 and 12 (as well as a few additional ones) are given in this appendix.

EQUATION FOR LOG-NORMAL LINES

$$\text{Estimated Number of Cycles} = \text{antilog } (\overline{\log N} + t_P \sigma_{\log N})$$

where:

$\overline{\log N}$ = average of the logarithms (to the base 10) of the observed N values,
$\sigma_{\log N}$ = standard deviation of the logarithms of the observed N values, and
t_P = Normal deviate corresponding to the cumulative percentage of failures divided by 100. For example:

P, per cent	t_P
5	−1.645
50	0
80	+0.842

FACTORS FOR SEVERAL DEFLECTION VALUES.

Deflection, in.	Hard Temper		Spring Temper	
	$\overline{\log N}$	$\sigma_{\log N}$	$\overline{\log N}$	$\sigma_{\log N}$
±0.540	5.083	0.050	5.235	0.043
±0.500	5.302	0.054	5.433	0.053
±0.460	5.549	0.070	5.637	0.046
±0.420	5.696	0.063	5.812	0.064
±0.380	5.894	0.068	5.998	0.070
±0.340 (2 runs combined)	6.133	0.080	6.866	0.565
run 1	...	...	7.106	0.521
run 2	...	...	6.625	0.501
±0.300 (2 runs combined)	6.885	0.489	...	...
run 1	6.754	0.362	...	...
run 2	7.062	0.540	...	...

EQUATION FOR LOG-LOG-NORMAL LINES

$$\text{Estimated Number of Cycles} = \text{antilog}\,[\text{antilog}\,(\overline{\log\log N} + t_P\sigma_{\log\log N})]$$

where:

$\overline{\log\log N}$ = average of the logarithms of the logarithms (to the base 10) of the observed N values,

$\sigma_{\log\log N}$ = standard deviation of the logarithms of the logarithms of the observed N values, and

t_P = Normal deviate (see Log-Normal).

FACTORS FOR SEVERAL DEFLECTION VALUES.

Deflection, in.	Hard Temper		Spring Temper	
	$\overline{\log\log N}$	$\sigma_{\log\log N}$	$\overline{\log\log N}$	$\sigma_{\log\log N}$
±0.360	...	...	0.800	0.019
±0.340 (2 runs combined)	...	...	0.835	0.036
run 1	...	...	0.850	0.033
run 2	...	...	0.819	0.032
±0.300 (2 runs combined)	0.837	0.030	...	...
run 1	0.829	0.022	...	...
run 2	0.845	0.034	...	...

EQUATION FOR EXTREME-VALUE CURVES

$$\text{Estimated Number of Cycles} = \frac{\Sigma a_i N_i}{k} + \left(\frac{\Sigma b_i N_i}{k}\right) y_P$$

where values of a_i and b_i are given on p. 72 of reference (**17**):

N_i = observed values of number of cycles to failure,

k = number of subgroups,

P = cumulative percentage of failures divided by 100, and

y_P = $-\log_e(-\log_e P)$; values may be read from Table 2 of "Probability Values for the Analysis of Extreme Value Data," National Bureau of Standards, Applied Mathematics Series 22, p. 19 (July 6, 1953).

FACTORS FOR SEVERAL DEFLECTION VALUES.

Deflection, in.	Hard Temper		Spring Temper	
	$\frac{\Sigma a_i N_i}{k}$	$\frac{\Sigma b_i N_i}{k}$	$\frac{\Sigma a_i N_i}{k}$	$\frac{\Sigma b_i N_i}{k}$
±0.540	115.396	12.492	164.901	12.946
±0.420	469.791	57.015	608.165	88.511
±0.340 (2 runs combined)	1 262.833	198.521	9 201.697	10 200.613
run 1	...	...	13 461.418	13 543.784
run 2	...	...	4 941.975	6 857.441
±0.300 (2 runs combined)	8 121.496	10 494.047	...	...
run 1	5 118.639	5 265.283	...	...
run 2	11 124.352	15 722.811	...	...

EQUATION FOR LOG-EXTREME-VALUE CURVES

$$\text{Estimated Number of Cycles} = \text{antilog}\left[\frac{\Sigma a_i \log N_i}{k} + \left(\frac{\Sigma b_i \log N_i}{k}\right) y_P\right]$$

where: (see Extreme-Value Curves).

FACTORS FOR TWO DEFLECTION VALUES.

Deflection, in.	Hard Temper		Spring Temper	
	$\frac{\Sigma a_i \log N_i}{k}$	$\frac{\Sigma b_i \log N_i}{k}$	$\frac{\Sigma a_i \log N_i}{k}$	$\frac{\Sigma b_i \log N_i}{k}$
±0.340 (2 runs combined)	...	...	6.631	0.408
run 1	...	...	6.844	0.490
run 2	...	...	6.418	0.326
±0.300 (2 runs combined)	6.660	0.370	...	...
run 1	6.583	0.248	...	...
run 2	6.737	0.493	...	...

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DISCUSSION

MR. FRANK A. MCCLINTOCK[1] (*presented in written form*).—The usefulness of these already interesting results would be increased by the presentation of data on the reproducibility of the machining and on the variability of the strip stock from side to side or from one length to another. Are such data available? Should further tests be contemplated, if the program were laid out in randomized blocks rather than completely at random one could use analysis of variance to study the importance of such factors directly from the main body of the data instead of resorting to supplementary tests. If these additional factors are of importance, then the reported distribution functions are a composite of several. The use of these distributions, for example, to predict that part of the size effect which is due to statistical causes, would depend on which factors were of primary importance in contributing to the scatter.

MR. H. E. FRANKEL[2] AND MR. J. LIEBLEIN[2] (*presented in written form*).—The authors are to be congratulated on the thoroughness of their work. Particularly noteworthy is the inclusion of their extensive data in sufficient detail for the benefit of those who wish to make further studies of the subject. There are, however, several points upon which clarifying comments can be offered.

The major attempt of the authors lies in fitting a P-N curve (probability distribution of number of cycles to failure) to the observed data. The three distributions—the log-normal, the extreme-value, and the log-log normal which are discussed and compared have certain advantages at different portions of the curve.

Contrary to the statement on page 1105, there are three types of extreme-value distributions, originally derived by Fisher and Tippett (**22**).[3] Each type is for a maximum, but the mirror-image can be made to serve for a minimum.

(*a*) Type I is the one utilized by the authors. As they point out, this distribution for minima is skewed to the left, but their data are skewed to the right; hence, this distribution does not seem to be applicable. Although the curve goes to infinity in both directions, this does not necessarily mean that the data from which maxima (or minima) are taken must also be unlimited.

(*b*) Type II is a distribution that is infinite in only one direction, having an upper limit, in the case of minima, and, therefore, skewed to the left. However, this particular distribution may not have as much interest, since it applies only to minima, for data which do not have a limit.

(*c*) Type III is skewed to the right and corresponds to minima for values having a lower limit, such as would be the case with the "strengths" of the flaws called for by the "weakest-link" theory. It is, therefore, suggested that perhaps this type commends itself as the one for fitting an extreme-value distribution to the data in place of the type I discussed by the authors. This type III distribution for minima is probably more familiarly known as the Weibull distribution.

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1123.

The authors have, apparently without realizing it, fitted a Weibull distribution to the data when they fitted a type I distribution to the logarithms as in Fig. 10. It has been shown that if data follow a Weibull distribution, then their logarithms follow a type I extreme-value distribution. For the higher stress levels, at any rate, this distribution is not improved upon by the log-normal. Since there is skewness present in the logarithms it may seem worthwhile to use a Weibull distribution. However, there may be a difficulty in the presence of a so-called "minimum life" that is, below which a specimen will have zero probability of failing; this minimum life will depend upon stress level. A more flexible form of this distribution is available having three parameters (**23**), which give it an important advantage over the two-parameter log-normal or type I extreme-value curves. The third parameter is the minimum life, mentioned above, and by suitably varying this parameter it may be possible to use a single distribution to fit all stress levels. If this can be done, it will give a more useful and satisfactory understanding and treatment of fatigue phenomena than trying, on a purely empirical basis, to fit two different, unrelated distributions to a continuous fatigue curve.

The log-log normal distribution apparently was introduced as an attempt to fit the data. Since it is an empirically obtained function there are no valid reasons for expecting the same distribution to fit any other situations.

MR. PHILLIPS WHIDDEN.[4]—I agree with the comments of Mr. Frankel and Mr. Lieblein. I want to add one more point.

I note that no attempt has been made by the authors to randomize the order of deflection levels. This leads to the result that on the *S-N* diagram, those higher deflection levels were all attained before a certain date, which was around February 1, 1953, and all those lower deflection levels after that.

This, of course, leaves room for real criticism of the procedure because of the obvious confounding of deflection levels with time.

MR. J. C. MILLSON[5] (*presented in written form*).—This paper by Miss Torrey and Mr. Gohn is of interest not only for the data it presents but because it will allow others to make use of the experimental results for their own statistical treatment.

The authors' mention that Fig. 8 is a graphic illustration of the change in distribution brought about by a change in deflection level. It has been the conclusion of our staff, working on the study of pitting corrosion and using the extreme value distribution for analyzing the results, that such a change in trend could also be the result of a particular experiment being out of control. The close agreement of log-normal, extreme value, and log-log normal curves is so good compared to many in the literature that this departure warrants examination.

The authors' long experience in this field may justify conclusion 2, purely from their knowledge of the material. It might also be said that the apparent lack of sensitivity at low deflections is due almost entirely to the fact that testing ended at 10^8 cycles, and that at the lower deflections (see Tables I and II) most of the specimens had not broken at this level. Hence, all that can be concluded is that the life is 10^8 or greater and running more samples cannot improve the estimate. Put rather differently, the standard deviation of one run of one temper at high deflections can be well estimated (see Table I) with 23 deg of freedom, but estimates at low deflections would have to be based on only 2, 3, or 4 deg of freedom.

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Two approaches to this problem are possible: (1) to agree that lives beyond 10^8 are of no interest and to ignore the low stress end of the curve; (2) if the low end contains information worth having, carry out the test with the same number of samples beyond 10^8 cycles.

The final curve, Fig. 15 (response curve) suggests a most interesting way of analyzing fatigue data, and the authors are to be congratulated for this excellent suggestion. Their particular curves are of limited range, and methods of extending the lines would be worth having.

MR. F. B. STULEN[6] AND MR. H. N. CUMMINGS[6] (*presented in written form*).—Reading and studying this paper in considerable detail has given us a great deal of satisfaction because in all of the findings and the conclusions of the authors we find confirmation of our own findings and conclusions. We have tested and statistically analyzed, to date, over 4000 R. R. Moore rotating-beam specimens, principally steel, of which nearly 3000 have already been reported in WADC Technical Reports references (**24,25,26**). Although we used a different metal, different type of specimen, different method of fatigue stressing, and different speeds of testing, we find ourselves in complete agreement with the three conclusions stated at the end of the paper.

In going through the paper we find a few places where we should like to make comments or ask questions. Regarding the sheet fatigue specimens, the ASTM Manual on Fatigue Testing[7] does not indicate any special treatment of the edges. Assuming that the flat strip specimens used by the authors had the sharp edges implied by the "cross-milling" procedure stated in the paper, it would be interesting to know if the bronze specimens showed any significant edge-weakness. A study of the location of the nucleus of fatigue failure might reveal such a weakness, if in general the fractures nucleated at or close to an edge. It has been shown (**27,28**), that steel flat specimens show this weakness, but so far as we know there are no statistical studies on the subject with reference to the non-ferrous metals. For design purposes, it would be useful to know whether or not rounding the edges of non-ferrous parts would significantly increase their fatigue strength.

As a matter of increasing our understanding of the mechanism of fatigue, it would be interesting to know if the authors have studied the fracture surfaces of the bronze specimens to see if the number of nuclei of fracture has any relation to the stress level. We found a clear correlation in the case of both air melted and vacuum melted high-strength steels (**24,26**), but have not yet had the opportunity to investigate non-ferrous metals at both low and high stress levels.

Under the heading "Data" the authors state that the "run test" indicated that in only one set of data was there indication that the two runs could not be treated as a single sample, although "further analysis showed that the data for three other deflection levels . . . . are in difficulty." We recommend another simple test, the "rank test," which is described by Wilcoxon (29). We found this rank test picked out not only the set of data that the run test detected, but also two of the other three sets, that were "in trouble." Of course, this one occurrence does not prove the rank test to be always more sensitive, but at least it suggests another simple way of catching differences in "populations."

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[6] Curtiss-Wright Corp., Propeller Div., Caldwell, N. J.

[7] "Fatigue Manual," (Issued as separate publication *ASTM STP No. 91*) (1949).

We were particularly interested in the plots, on normal probability paper, of life at constant stress (deflection), Figs. 11 and 12, and of strength at constant life, Fig. 15, to note another confirmation of our observations on high-strength steel. The slopes of the straight lines drawn through the sets of test points increase as the stress decreases, on Figs. 11 and 12, and as the life increases, on Fig. 15. In other words, the scatter is not constant for all values of stress, nor for all values of life, in the case of the bronze tested by the authors. The scatter in life is relatively large at low fatigue stress, and the scatter in strength is relatively large at long fatigue life. It seems to us that this characteristic, relatively high variability in the lower stress, longer life, region, which we found (24, 25,26), in steel, and the authors have found in at least one non-ferrous metal, may be tied in somehow with the mechanism of fatigue. There may be a change in the sensitivity to whatever stress raisers cause crack initiation and propagation, as the fatigue stress level is raised. Metallographic examination of the fracture surfaces might suggest a hypothesis as to the reason for this change in population.

The authors state in their conclusions that what they refer to as "response" curves have the advantage that change in the type of distribution of fatigue life for small *versus* large deflection levels does not seem to have any appreciable effect. And they recommend that the use of response curves for several values of cycle life be investigated further. In effect, they recommended investigating the analysis of fatigue data on the basis of probabilities of surviving various stresses for fixed numbers of life cycles. It is interesting to note that, until very recently, the S-N curves reported as a result of fatigue testing have actually been hybrids. That is, the high stress part of an S-N curve was drawn through the mean, or the median, life (or log-life) values, and the lower part, below the knee, was drawn through either the average stress, or sometimes conservatively below the stress at which the last specimens failed. To put it in the words of the mathematicians, for the upper part of the graph, stress was the independent variable, and, for the lower part, the fatigue life, which had been the dependent variable became the independent variable. Actually, so far as we have been able to determine in our own studies, this was not particularly serious so long as only one curve—the average curve—was drawn. But for probabilities other than 50 per cent, the variables are not interchangeable, in general, excepting that where the graph is a straight line and the variance is practically constant along the straight line we have found (24) that for all practical purposes they may be interchanged. To present S-N curves of constant probability that are consistent throughout the whole range of life cycles, we have analyzed all our data for the mean and the standard deviation of stress at constant life. And, exactly as suggested by the authors, we have used the response testing techniques for the long-life portions of the curves, following Finney's "Probit Analysis"

(30). (For the short-life portions of the curve we use the standard linear regression analysis selecting sections of the curve that can reasonably be represented by linear functions.)

In closing, we wish to compliment the authors on having presented such a comprehensive study of a problem so fundamental to the use of statistical methods in the analysis of fatigue test data.

MR. E. H. SCHUETTE[8] (*presented in written form*).—The authors are to be thanked for having prepared a collection of test data specifically designed for checking today's hypotheses regarding the nature of life distribution. Of particular interest is the distinct change in this distribution that occurs at the breakpoint in the S-N diagram.

In most aluminum and magnesium alloys, no such sharp break in the S-N curve is observed. Instead, the curve shows a continuous decrease in slope throughout the life range usually recorded. It has been my own casual observation—never so rigorously checked as in this paper—that the nature of the distribution of lives also changes gradually with changing stress or deflection. Thus, for our purposes, a good fit will not be obtained by any one, two, or other finite number of distribution forms. To me, this further emphasizes the desirability of using medians rather than means, and other non-parametric methods for estimating extremes, so that results will be unaffected by differences in the nature of life distribution.

I see no need for precise definition of the tails of the distribution; it seems completely pointless to me to worry over a stress difference of perhaps 0.1 per cent when the very next batch of supposedly identical material may turn up with properties different by several per cent.

MR. G. W. STICKLEY.[9]—I would like to ask how the curves are fitted to the data and what equations were used for the curves representing the different directions.

MISS M. N. TORREY (*author*).—It seems convenient to answer the discussions in reverse order.

In answer to Mr. Stickley, the equations for all the fitted curves are given in the Appendix to the paper.

Mr. Whidden's suggestion for randomizing the order of the deflection levels is a good one. It is a procedure which will be used in the next experiment. However, in the spring temper data (shown in Table II) the two runs for the deflection level ±0.360 in. were made about a year after all other runs were finished. The observed values appear to fit in between the values for the deflection levels ±0.380 in. and ±0.340 in. as well as if the levels had been tested in order. This gives some assurance that the marked increase in variability at the lower deflection level was not due to a change in the machine.

This relates also to one of Mr. Millson's comments. He suggested that the increase in variability could be an indication of an experiment being out of control. The consistency of the data for ±0.360 in., mentioned previously, seems to indicate that the experiment was in control. In the next experiment the order of the deflection levels will be randomized so that the change in level is not confounded with time.

Concerning other comments by Mr. Millson about computing standard deviation values for the distributions of cycle life at the several deflection levels, the standard deviations would not be computed in the usual way when there are run-outs. A standard deviation value might be estimated from a regression line fitted to the cumulative distribution, or the method of estimating the standard deviation of a truncated distribution might be used if the proportion of runouts were small. Either method would, in effect, use the results for the entire sample.

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[9] Assistant Chief, Mechanical Testing Div., Alcoa Research Labs., New Kensington, Pa.

In answer to Messrs. Frankel and Lieblein, the authors used the term "ultimate extreme value distributions" to refer to the distributions for minima and maxima, not to refer to the types of extreme value distributions. (The word "ultimate" was suggested by Dr. John Tukey.)

One difficulty with using the type III extreme value distribution is the necessity for estimating three parameters. The usual small samples are hardly adequate for estimating two parameters; their use for estimating "minimum life, below which a specimen will have zero probability of failing" would seem to be impractical except for the case where the value of minimum life is estimated to be zero.

Mr. McClintock raises interesting questions on the reproducibility of the machining and on the variability of the strip stock. Such information is not available for this experiment but it is planned to get such information in the next experiment.

There is one difficulty in using analysis of variance techniques where the data cover a range of deflection levels; that is the choice of a transformation that will normalize the various distributions of cycle life. In such a study it might be necessary, therefore, to restrict the analysis to a limited range of deflection levels.

MR. G. R. GOHN (*author*).—Mr. Cummings has raised several questions pertaining to the actual testing. We have not observed any edge effect in our tests. The specimens tested had a nominal thickness of 0.040 in. From the time the first crack was detected in the "as rolled" surface, rate of crack propagation was so fast that it was very difficult to stop the test run in time to make a metallographic examination prior to complete fracture. No studies have been made in these tests to determine the location of the fatigue nucleus. Perhaps this can be included in the next series of tests, in which case a better answer can be given to Mr. Cummings' question. His suggestion with respect to the possibility of rounding off edges is a good one and an experiment will be performed on several sets of specimens, half with rounded edges, to see if the fatigue life of the specimens with rounded edges is different from that of specimens prepared by the normal cross-milling methods.

The results of the "rank test" are interesting. There are possibly many other ways of analyzing the data. In our opinion, the most useful thing accomplished in this paper is the presentation of new data for others to study.

M. N. TORREY AND G. R. GOHN (*authors' closure*).—The authors are pleased by the interest shown in the paper and are appreciative of the several suggestions for future investigations.

Since the presentation of the paper, a study has been made of the appropriateness of the log-extreme-value distribution for fitting the observed distributions of cycle life for the higher deflection levels. This study was prompted by the recommendations of Messrs. Frankel and Lieblein that the type III extreme value distribution might fit the data for all stress levels. They indicated that fitting a type I distribution to the logarithms of the observed values is analogous to fitting a type III distributions to the observed values.

Preliminary results, using the data for hard-temper strip, deflection levels ±0.540 in. and ±0.340 in. indicate:

1. The log-normal distribution fits

best (P = 0.56 and 0.83 respectively),

2. The extreme-value distribution is next best (P = 0.07 and 0.67 respectively), and
3. The log-extreme value distribution is the poorest (P = 0.04 and 0.31, respectively).

P = probability of as great a difference between the observed and theoretical distributions due to chance alone.

The number of degrees of freedom in all cases is 6 which is small due to the grouping of the values in the tails. As mentioned in the paper (p. 1107) it would take perhaps hundreds of observations to show which distribution is actually best. However, it does appear that the log-normal distribution may be used for fitting data at the finite life end of the deflection-cycle life curve as concluded in the paper on p. 1109.

Three comments are offered on the discussion by Messrs. Stulen and Cummings:

1. In Fig. 15 of the paper, the slopes of the lines appear to increase as life decreases (not increases). Actually there are not enough points to determine whether the slopes change or not.

2. The lines in Fig. 15 are regression lines relating percentage of failures and deflection level. They are not intended to represent cumulative distributions of deflection levels or strength. Thus they do not give estimates of "scatter in strength."

3. The authors believe that in the usual type of fatigue testing, stress is always the independent variable and that fatigue life is always the dependent variable. They are not "interchangeable."

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A WIRE FATIGUE MACHINE FOR INVESTIGATION OF THE INFLUENCE OF COMPLEX STRESS HISTORIES*

By H. T. Corten[1] and G. M. Sinclair[1]

Synopsis

A wire fatigue testing machine is described that operates on the principle of a deflected rotating strut. This equipment was designed to investigate fatigue life for complex stress histories, particularly those consisting of repeated blocks of cycles. With this wire machine the stress amplitude may be changed rapidly from one level to another without inducing noticeable vibration in the wire specimen. For a comparative study of material behavior, wire fatigue testing incorporates several particularly desirable features, namely, inexpensive specimens and small scatter of data. The results of constant stress amplitude tests of steel wire are presented and analyzed statistically.

In the field of progressive fracture of metals, knowledge has accumulated to the point where it is recognized that numerous factors influence fatigue behavior. Most of the early work, however, must be viewed as exploratory in view of the statistical variability of results, a fact that has been fully recognized for only a few years. When quantitative results are desired, as for example a comparison of the fatigue strength or fatigue life of members subjected to different stress histories, a statistical study including experimental design and analysis of the results becomes imperative. Possibly the greatest benefit to be derived from such a procedure is an estimate of the exactness that can be associated with a given result, or, as is more often the case, the degree of inexactness that must be accepted.

The reliability of a measurement may be increased by employing a larger number of specimens; however the estimate of reliability, say 95 per cent confidence limit, varies inversely as the square root of the number of specimens.[2] For example, if 10 specimens were used to measure a result and it was desired to reduce the range enclosed by the 95 per cent confidence limits to approximately one-half of their present value, a total of 37 specimens would be required. Considering the time and cost associated with testing this number of specimens, a point of diminishing returns is soon reached, unless very simple, rapid testing procedures and inexpensive specimens can be employed. Consequently, for problems involving what might be termed "fundamental material behavior" in which the results of identical specimens are to be compared, wire specimens and appropriate testing machines appear to possess many advantages.

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[2] Actually varies as the square root of $n - 1$ where n is the number of specimens.

Purpose

In this paper a wire fatigue testing machine is described that was designed to facilitate the study of the influence of stress history on fatigue strength and fatigue life. The basic design is sufficiently versatile that it may be readily adapted (*a*) to progressively increasing or decreasing load histories, (*b*) to a simple prestress followed by test stress loading, or (*c*) to a variety of periodically repeating load histories. The intended future application of the machine is the determination of the effect of loading history, consisting initially of repeated blocks of completely reversed stress cycles, on fatigue life. During each block a specimen will be subjected to reversed cycles of a high stress for a predetermined percentage of the block and the remainder will be applied at a lower stress.

In the paper the design and operation of the wire machine are considered, and only data obtained during *constant* stress amplitude operation are presented.

Theoretical Considerations and Design

The use of wire specimens requires that the region of maximum stress be located away from the points where the specimen is gripped or loaded. A deflected rotating strut loaded with axial compressive forces is a commonly employed arrangement (**1, 2**)[3] that possesses the desired characteristics and for which the maximum stress may be accurately computed if the behavior of the strut or wire is maintained elastic. However, in those instances where this arrangement has been employed the process of changing from one stress level to another necessitates movement or rotation of parts that possess large inertia in comparison to the specimen generally used to determine the deflected configuration. Thus rapid changes of stress from one level to another are not feasible with the available machines.

The machine described in this paper employs a deflected rotating strut specimen; however one end of the specimen, used as the drive end, is fixed rigidly. The other end of the specimen is free to rotate (in the plane of bending) and follows a curved path such that the fixed (drive) end of the specimen is theoretically subjected to zero moment. Since both ends of the specimen sustain zero moment the behavior of the specimen is essentially that of a pivot-ended deflected strut, and the computation of stress is based on this condition. The movable end of the specimen fits into a miniature ball bearing and housing which is free to rotate and assume the configuration imposed by the specimen.

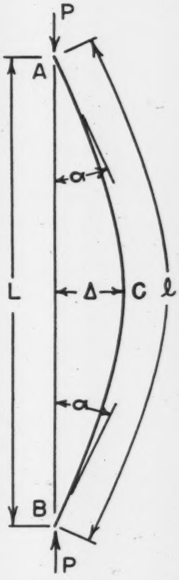

Fig. 1.—Deflected Configuration of Elastic Strut.

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1132.

By maintaining the inertia of this assembly at a small value, rapid changes of stress are possible without inducing appreciable vibration. The miniature bearing housing is pinned into a small trolley that moves on a curved track. The curved track theoretically imposes the appropriate shortening of the distance between the ends of the specimen to insure a zero moment at the fixed end at various deflected configurations.

TABLE I.—VALUES OF DIMENSIONLESS RATIOS FOR COMPUTING STRESS AND DEFLECTED SPECIMEN LENGTH.

α, deg	$\frac{P\ell^2}{EI}$	$\frac{\Delta}{\ell}$	$\frac{\ell - L}{\ell}$
2	9.87111	0.01111	0.000304
4	9.87562	0.02221	0.001218
6	9.88315	0.03330	0.002741
8	9.89369	0.04436	0.004944
10	9.90728	0.05538	0.007603
12	9.92391	0.06636	0.010939
14	9.94366	0.07729	0.014879
16	9.96648	0.08816	0.019417
18	9.99244	0.09898	0.024547
20	10.02157	0.10971	0.030270
22	10.05390	0.12035	0.036576
24	10.08948	0.13091	0.043466
26	10.12836	0.14136	0.050931
28	10.17059	0.15171	0.058966
30	10.21623	0.16195	0.067568
32	10.26535	0.17206	0.076786
34	10.31798	0.18204	0.086435
36	10.37425	0.19188	0.096690
38	10.43420	0.20158	0.107479
40	10.49794	0.21112	0.118797
42	10.56554	0.22051	0.130633
44	10.63711	0.22972	0.142981
46	10.71275	0.23876	0.155831

A deflected strut subjected to zero end moment is shown in Fig. 1. There is associated with each value of the deflected length L a particular value of the angle α. The exact relation between the load P; the maximum deflection, Δ; L; the modulus of elasticity, E; the moment of inertia, I; the undeflected length, ℓ; and α for large deflections is given by Southwell (3). The results may be conviently expressed in terms of the dimensionless ratios $\frac{P\ell^2}{EI}$, $\frac{\Delta}{\ell}$, and $\frac{\ell - L}{\ell}$. Values of these dimensionless ratios for various values at α are given in Table I. The maximum stress in the specimen is given by

$$S = \frac{P}{A} + \frac{Mc}{I} \qquad \text{(1)}$$

where $M = P\Delta$ and A is the area of the cross-section. In terms of the dimensionless ratios, the stress is given by

$$S = \left(\frac{P\ell^2}{EI}\right)\frac{EI}{\ell^2 A} + \left(\frac{P\ell^2}{EI}\right)\left(\frac{\Delta}{\ell}\right)\frac{Ec}{\ell} \qquad \text{(2)}$$

For the relative specimen dimensions commonly employed, the first term is usually negligible. To investigate this condition, it is convenient to divide the stress S_P due to the axial load by the total stress due to axial load and bending moment $(S_P + S_M)$. Thus

$$\frac{S_P}{S_M + S_P} = \frac{1}{4\left(\frac{\ell}{c}\right)\left(\frac{\Delta}{\ell}\right) + 1} \qquad \text{(3)}$$

In the present machine, $\frac{\ell}{c} = 400$ and the range of angles α that have proved usable is from 14 to 26 deg. At $\alpha = 14$ deg the value of the ratio from Eq. 3 is 0.0081 and at $\alpha = 26$ deg the value is 0.0044. Since the error over the useful range of α is considerably less than 1 per cent, the stress has been computed by

$$S = \left(\frac{P\ell^2}{EI}\right)\left(\frac{\Delta}{\ell}\right)\frac{Ec}{\ell} \qquad \text{(4)}$$

The point of maximum stress occurs theoretically at the center of the deflected strut for all values of angle α.

The track which controls the deflected length of the strut may be laid out from the data in Table I. Figure 2 shows schematically the track layout, motor location and deflected strut specimen and Fig. 3 shows the wire machine fitted with the additional devices necessary to alter the load amplitude periodically.

The theoretical layout of the machine was readily accomplished, but the accurate manufacture and assembly of the basic elements of the machine (the curved track and motor assembly) presented greater difficulty. The deflected length of the specimen changed very little for small angles of α and in no case exceeded 0.6 in. in the useful stress range. Consequently, to achieve the desired zero moment end conditions, the track must be made and assembled such that the pivot point on the trolley that holds the movable end of the specimen could be positioned accurately within several thousandths of an inch and several minutes of a degree with respect to the fixed end of the specimen.[4]

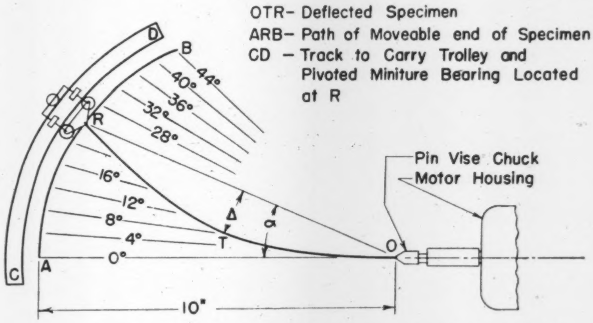
OTR- Deflected Specimen
ARB- Path of Moveable end of Specimen
CD - Track to Carry Trolley and Pivoted Miniture Bearing Located at R
Pin Vise Chuck
Motor Housing
D
B
44°
40°
36°
32°
28°
R
16°
12°
8°
4°
0°
Δ
α
T
O
C
A
10"

Fig. 2.—Theoretical Layout of Wire Fatigue Machine.

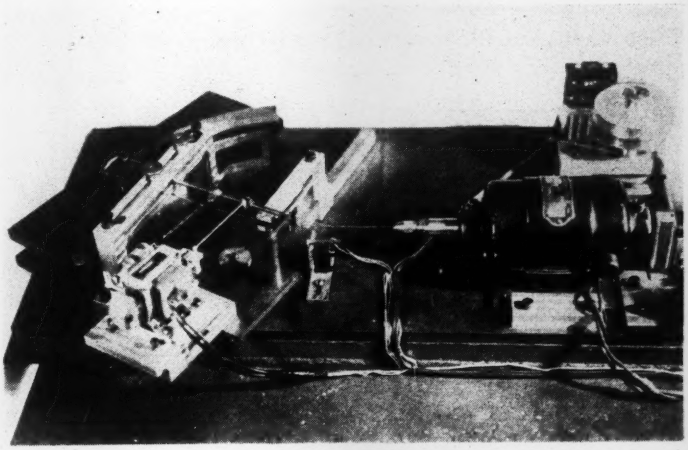

Fig. 3.—Wire Fatigue Machine Equipped for Fluctuating Load Experiments.

To achieve accuracy that could be verified experimentally, a track consisting of a circular arc was substituted for the theoretically specified track. The location of this track could be verified experimentally after assembly because it possessed a fixed known center of curvature from which the motor and chuck assembly could be accurately located. This procedure afforded a simple assembly for which the difference between the theoretical and actual track could be computed. A trial and error procedure led to a track radius and center for which the difference between the ideal track and actual track was very small over the usable stress range. Figure 4 shows the layout and dimensions employed in the present machine. Figure 5 shows the computed difference between the theoretical and actual track for various values of α. The maximum difference is 0.0075 in. at $\alpha = 24$ deg. The maximum percentage error in the difference between the undeflected and deflected lengths, $\ell - L$, occurs at the small values of α and is 3.1 per cent at $\alpha = 10$ deg, 2.7 per cent at $\alpha = 16$ deg, 1.7 per cent at $\alpha = 24$ deg, and decreases rapidly thereafter as α increases.[4] The track is exact at $\alpha = 0$ deg and again at approximately $\alpha = 32$ deg.

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[4] If the maximum and minimum usable stresses are known beforehand, the track may be arranged to give a minimum error in this range. The present machine was designed for use in a stress range somewhat higher than is possible with the wire specimens employed.

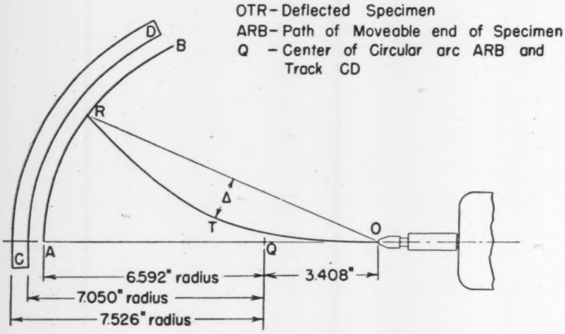
OTR - Deflected Specimen
ARB - Path of Moveable end of Specimen
Q - Center of Circular arc ARB and Track CD
D
B
R
Δ
T
O
C
A
Q
6.592" radius
3.408"
7.050" radius
7.526" radius

FIG. 4.—Layout of Circular Arc Track for Wire Fatigue Machine.

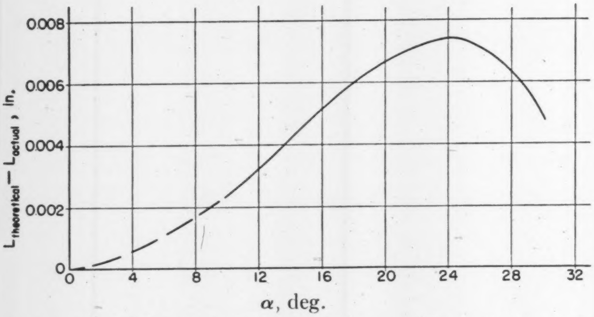

FIG. 5.—Difference in Length of Deflected Specimen Between Theoretical and Circular Arc Tracks.

The difference between the theoretical and actual track creates a condition in which the bending moment at the end of the specimen that is clamped in the motor chuck is not exactly zero. This moment is small and introduces no practical difficulties in the operation of the machine. Since the machine was designed for investigations in which the life or strength as measured by different sets of data obtained on the same machine would be compared, no error is introduced into the data or results. The computation of the absolute magnitude of the stress is the only quantity that is rendered slightly inaccurate as a result of the difference between the ideal and actual track. An analysis of the actual deflected configuration of the specimen and the absolute magnitude and location of the maximum stress is presented in the Appendix. Based on this analysis and a comparison between the location of maximum stress and the experimental fracture locations, it was concluded that the stress was increased by approximately 4 per cent due to the end moment that was introduced at the fixed end of the specimen. Since the absolute magnitude of stress was necessary only to insure that the behavior of the specimens was elastic, this absolute accuracy was deemed adequate. Equation 4 based on the ideal track gives accurate results with regard to relative magnitudes of stress. Consequently for purposes of comparison and presentation of results, Eq 4 was used to compute the stress.

EXPERIMENTAL DATA AND RESULTS

Fatigue life and endurance limit data from constant stress amplitude experiments are presented here to illustrate the experimental results obtained with the wire machine. The wire specimens used had a diameter of 0.050 in. and a length of 10 in. between pivot points. The over-all specimen length was approximately 11 in. Five thousand feet of hard-drawn brite basic steel wire, straightened and in 3-ft lengths was procured. The material was treated to prevent corrosion and stored in a dry tight container. As required, twenty to thirty 3-ft lengths were removed and cut to the proper specimen length.

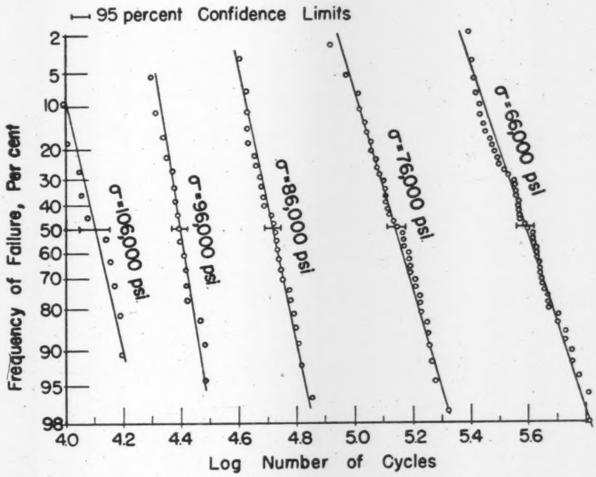

FIG. 6.—Logarithmic-Normal Frequency Diagram Showing Individual Fatigue Lifetimes Obtained at Different Stresses.

The tensile properties of the wire, obtained by wrapping the ends around 1-in. diameter loading pins were: yield strength at 0.2 per cent offset, 128,000 psi and tensile strength, 150,000 psi. The modulus of elasticity measured over a 10-in. gage length was $E = 30,000,000$ psi. The surface of the specimens, when examined under a microscope at 100 ×, looked scratched, with many of the scratches running in a circumferential direction. The specimens were used in the as received condition since all of the specimens that were examined exhibited essentially the same surface condition.

TABLE II.—FINITE LIFE FATIGUE RESULTS.
Based on Logarithmic-Normal Frequency Distribution.

Stress, psi	Number of Specimens	Mean log N	95 per cent Confidence Limits on Mean log N	Standard Deviation, σ	90 per cent Confidence Limits on σ
66 000	50	5.5864	5.5552 to 5.6176	0.1087	0.0940 to 0.1311
76 000	37	5.1396	5.1080 to 5.1713	0.0936	0.0803 to 0.1180
86 000	26	4.7146	4.6867 to 4.7425	0.0677	0.0563 to 0.0903
96 000	17	4.3937	4.3659 to 4.4215	0.05246	0.0422 to 0.0766
106 000	10	4.1021	4.0472 to 4.1571	0.0729	0.0568 to 0.1280

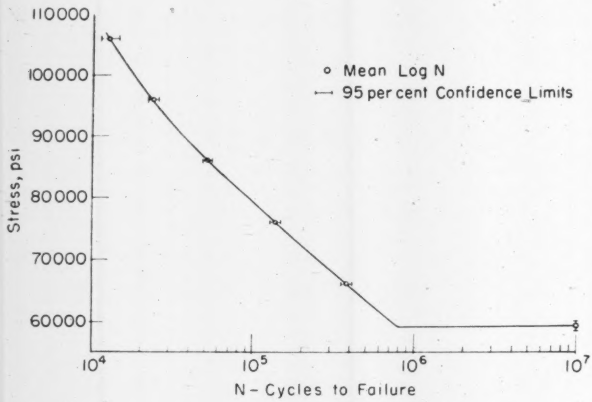

FIG. 7.—Stress-Cycles to Failure Diagram.

A few preliminary tests were made to locate the S-N curve and observe the behavior of the wire specimens at various speeds. A speed of approximately 6500 rpm was employed because it appeared to introduce the least vibration. A fork guide was added to prevent excessive vertical vibration of the specimens. At the speed chosen, the wire specimens exhibited only a slight tendency to vibrate in the vertical direction and no observable vibration was present in the horizontal direction or plane of bending. When an initially bent specimen was encountered the vibration increased but these specimens were discarded.

The fatigue lives, N, for the wire specimens are plotted in Fig. 6 in the form of a probability of failure *versus* log N diagram. Because of the availability of simple methods of computing confidence limits, the assumption of a log-normal life distribution was employed to analyze the data. The results of this analysis are given in Table II which also indicates the number of specimens tested at each stress level. Larger numbers of specimens were tested at the lower stress levels in an attempt to maintain the confidence limits for mean log N within a small constant value, approximately ±0.03, at all stress levels. The solid straight lines in Fig. 6 are based on the assumption of a log-normal life distribution and are drawn through the points obtained from the calculated parameters, mean log N and the standard deviation, σ.

It should be noted from the steep slopes of the lines in Fig. 6 that the

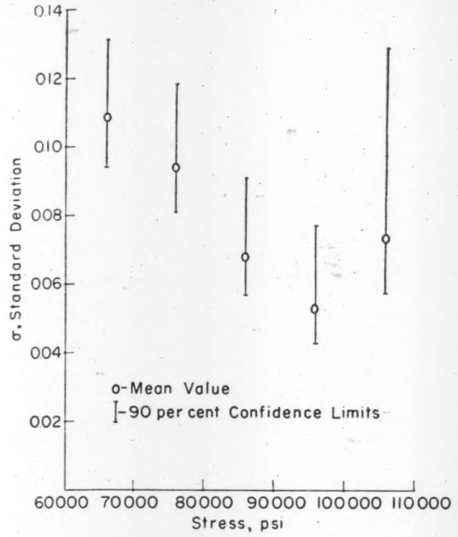

FIG. 8.—Standard Deviation at Various Stresses.

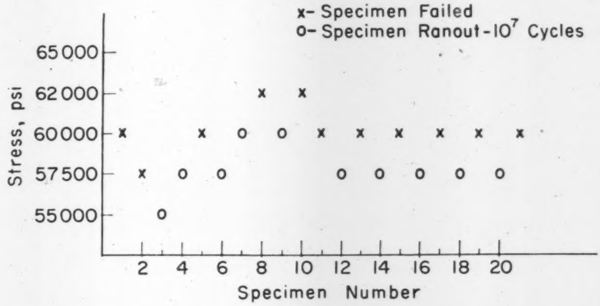

FIG. 9.—Failure-Runout Diagram of Staircase Statistical Analysis for Endurance Limit.

scatter of life at a given stress was relatively small in all cases. This characteristic has been observed in other studies of wire, particularly steel wire (4) and is apparently due, at least in part, to the large proportion of the surface of the wire that is subjected to (or near) peak stress. Accordingly, the values of the standard deviation are small and the confidence limits for mean log N are narrow, a characteristic that is particularly desirable in comparison studies.

In Fig. 7, an S-N diagram is presented showing values of mean log N and the 95 per cent confidence limits for the finite life region. It is evident that a smooth nearly-straight S-N curve was obtained for the range of stress levels employed. At a stress of 106,000 psi, the life was approximately 10,000 cycles; this stress was slightly above that at which deviation from linearity was noticeable on the static stress-strain curve. Consequently, no higher levels of stress were explored, and, due to inelastic bending, some question may exist about the accuracy of the stress computation of 106,000 psi. At a stress of 96,000 psi and below, the behavior was elastic and the accurate computation of stress was possible.

The variation of the standard deviation with stress is shown in Fig. 8. The standard deviation, σ, tends to decrease as the stress increases, a trend that is in general agreement with previous data (5). The large value of σ at a stress of 106,000 psi may be caused by the fact that the amount of plastic bending varied slightly between specimens, or it may be a distortion created by plotting the data for very short lifetimes in terms of log N.

In addition to the finite life data, the endurance limit was investigated by the staircase method of testing (6). Twenty-one specimens were employed with a stress increment of 2500 psi between steps. When a specimen sustained 10^7 cycles without fracture, it was considered to have runout and the test was discontinued. This study is shown in Fig. 9, and the statistical analysis gives:

Mean endurance limit, psi..	59 000
Standard deviation, psi.....	480
Standard error in estimate of mean, psi..............	460
Estimate of 95 per cent confidence limits for mean endurance limit, psi......	58 100 to 59 900

In general, the stress increment between steps should be from one-half to two times the standard deviation (6). Since approximately five times the standard deviation was employed as the increment, these data are not suited for close accuracy by the staircase analysis. There is no question that the mean endurance limit is between 57,500 psi and 60,000 psi; however, the values of mean endurance limit, standard deviation, and confidence limits may not be of high precision. As the accuracy of the endurance limit was not of importance in the immediate work contemplated for the machine, the preliminary results have been shown on the S-N diagram in Fig. 7.

SUMMARY

A wire fatigue testing machine is described that was designed especially for an investigation of the influence of complex stress histories on fatigue life. The major difference between this and previous wire machines is that the stress may be changed rapidly from one level to another without inducing noticeable extraneous vibration in the specimen. The obvious advantages of wire fatigue testing (that is, inexpensive specimens and short specimen set-up time) are combined with desirable characteristics of wire fatigue results. Small scatter of both life and endurance limit data obtained in wire testing provide a machine that is well suited for an experimental investigation of the influence of fluctuating stress histories employing statistical methods. Constant stress amplitude life and endurance limit data are presented to illustrate the performance of the machine and the statistical nature of fatigue data.

Acknowledgment:

This investigation was conducted in the research laboratories of the Department of Theoretical and Applied Mechanics as part of the work of the Engineering Experiment Station, University of Illinois, in cooperation with the Office of Naval Research. The advice and criticism of T. J. Dolan, Head of the Department, is greatly appreciated. Acknowledgment is due to Dean Bailey for his care in constructing the machine, and to G. L. Lovestrand, D. C. Bailey, and W. B. Whitler who assisted in various phases of the project.

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APPENDIX

ESTIMATE OF ERROR IN STRESS DUE TO CIRCULAR ARC APPROXIMATION OF THE THEORETICAL TRACK

From the theory of elasticity, the deflected length L of the specimen was computed for various angles α (Fig. 1). The values are listed in Table I in dimensionless form. The difference between the theoretical deflected length of the specimen and the actual deflected length based on the circular arc approximation is shown in Fig. 5 for various values of α. Over the range of stress levels employed, the actual track produced a deflected length that was from 0.0052 to 0.0075 in. shorter than the theoretical length.

The effect of this shortening on the maximum stress and location of the maximum stress may be estimated as follows: For each deflected length there is one value of the angle α for which the moment at each end of the specimen is zero as shown in Fig. 1. If the angle is different from the theoretically computed angle at one end due to end restraint (one end fixed in the chuck), a bending moment is introduced that produces a deflected curve similar to that shown in Fig. 10. The end loads P act parallel to the axis AE and form a couple $P\delta_1$ that is equal to the moment M. This configuration with an effective length AE shifts the point of maximum stress to point D and changes the magnitude of the maximum stress.

To estimate the maximum stress and its location let the deflected curve be approximated by a sine curve with reference axis AE. In Fig. 10, the angle α' is imposed on the specimen by the fixed chuck at end B. End A is free to rotate and makes an angle $\alpha_1 + \lambda$ with the line AB. The length $L_{\text{eff.}}$ and angle α_1 are required.

From Fig. 10 and the assumed sine curve:

$$\delta = \Delta \sin \frac{x}{L_{\text{eff.}}}\pi \;\ldots\ldots\ldots\ldots (5)$$

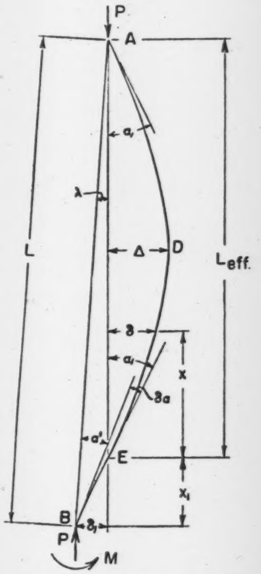

FIG. 10.—Deflected Configuration of Elastic Strut Subjected to Axial Load and End Moment.

The angle between the tangent to this curve at the distance x from the point E and line AE is given by:

$$\frac{d\delta}{dx} = \frac{\Delta\pi}{L_{\text{eff.}}}\cos\frac{x}{L_{\text{eff.}}}\pi \;\ldots\ldots\ldots (6)$$

Since the segment BE is itself a portion of another sine curve identical to ADE, the deflection δ and slope $\frac{d\delta}{dx}$ will be the same for the segment BE as for ADE at $x_1 = x$. Thus the angle $\delta\alpha$ is given by the difference of the slopes at $x = 0$ and $x = x_1$.

$$\delta\alpha = \left(\frac{d\delta}{dx}\right)_{x=0} - \left(\frac{d\delta}{dx}\right)_{x=x_1}$$

or

$$\delta\alpha = \frac{\pi\Delta}{L_{\text{eff.}}}\left(1 - \cos\frac{x_1}{L_{\text{eff.}}}\pi\right) \ldots\ldots (7)$$

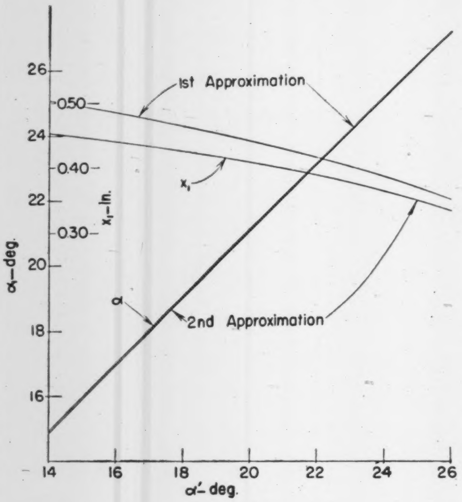

FIG. 11.—Computed Values of α_1 and x_1 for Various Values of α' for Circular-Arc Track.

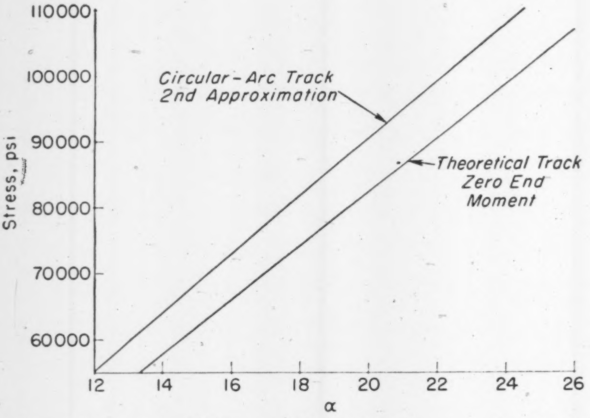

FIG. 12.—Comparison of Computed Values of Stress for Theoretical and Circular-Arc Tracks.

With an appropriate value of $\delta\alpha$, the distance x_1 may be computed from Eq 7. For a given angle α and deflected length, L, the rate of change of α with L (approximate) may be obtained from the data in Table I. The average slope for each 2-deg. interval was computed and this value was plotted at the midpoint of the interval. Thus for a given value of α_1 and an error δL in the deflected length L of the specimen, the difference, $\delta\alpha$, between α_1 and α' is given by:

$$\delta\alpha = \left(\frac{\delta\alpha}{\delta L}\right)_{\alpha_1} \delta L \qquad (8)$$

The exact evaluation of $\delta\alpha$ from Eq 8 is not possible because values of δL are known for various values of α' but not for α_1. The angle α_1 will be somewhat larger than α' but the exact difference is not known. By a method of successive approximations a sufficiently accurate value of $\delta\alpha$ may be obtained. The angle $\delta\alpha$ is first computed using the values of $\frac{\delta\alpha}{\delta L}$ and δL evaluated at α'. From this first approximation of $\delta\alpha$, approximate values of x_1, δ_1, λ, and α_1 may be computed. Based on this approximate value of α_1, a second approximation of $\delta\alpha$ may be computed. The process may be repeated as many times as is necessary to obtain accurate values of $\delta\alpha$, x_1, δ_1, and λ. The results of the second approximation were sufficiently accurate in view of the initial assumptions upon which the analysis was based. Because of the nature of the approximations, the first approximation slightly overestimated the values of $\delta\alpha$, x_1, λ, and α_1 and the second approximation slightly underestimated these values. However, when the results for a third approximation were considered, the difference between the second and third approximation were found to be negligible. The results of the computation of α_1 and x_1 corresponding to various values of α' are given in Fig. 11.

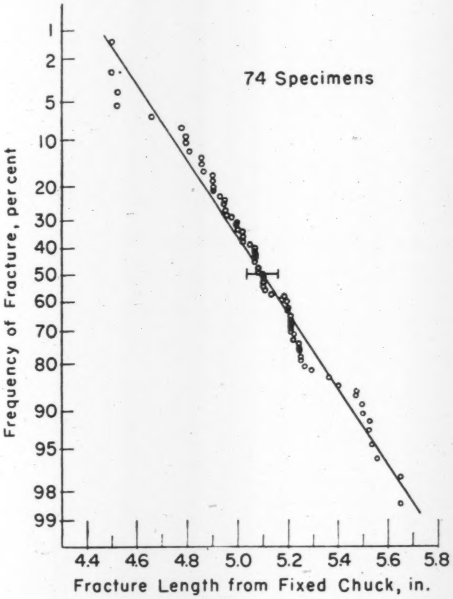

FIG. 13.—Fracture Length Diagram Showing Mean Fracture Length and 95 per cent Confidence Limits.

The stress in the wire specimens is given by Eq 4 as:

$$S = \left(\frac{P\ell^2}{EI}\right)_{\alpha_1}\left(\frac{\delta}{\ell}\right)_{\alpha_1}\frac{Ec}{\ell_{\text{eff.}}} \; \ldots\ldots \; (9)$$

in which the dimensionless ratios are evaluated for α_1 and the value of $\ell_{\text{eff.}}$ is computed from the value of x_1. The stress in the specimen based on an ideal track and for the circular-arc approximation of the track are compared in Fig. 12 for various values of α'. According to the computations the circular arc approximation shown in Fig. 4 causes the stress to be from 9 to 11 per cent higher than the stress associated with an ideal track.

To confirm this analysis it is of interest to compare the measured length from the chuck to the point of fracture with the computed distance to the point of maximum stress. The experimental data for 74 specimens are presented in Fig. 13 in a frequency diagram. If the fracture lengths were normally distributed about a mean value, the data in Fig. 13 should result in a straight line; the data apparently form a fair straight line. A stress of 99.0 per cent of the maximum occurs at a distance of 0.445 in. from the point of maximum stress. Thus 67 of the 74 specimens broke at points where the stress was between 99 and 100 per cent of the maximum value. Based on the assumption of a normal distribution, the mean value and 95 per cent confidence limits for the fracture length were computed and are also shown in Fig. 13. The mean value is 5.101 in. with confidence limits from 5.036 to 5.166 in. This must be compared with the computed values of 5.23 in. at $\alpha' =$ 16 deg and 5.20 in. at $\alpha' = 24$ deg. A 5.00-in. length represents the exact center of the specimen. The measured length indicates a shift in the location of maximum stress of approximately one-half of the computed value. This indicates that due to small errors in the manufacture and assembly of the machine, the value of the actual stress is between those values given in Fig. 12 for the ideal track and circular arc track. Consequently the lines in Fig. 12 representing Eq 4 and Eq 9 may be considered as upper and lower limits for the actual stress in the wire specimens. Based on the measured fracture lengths, the stress is approximately 4 per cent higher than the value computed from Eq 4, and a range of from 1 to 7 per cent higher is indicated by the 95 per cent confidence limits on the fracture length.

DISCUSSION

Mr. G. R. Gohn.[1]—The authors are to be congratulated upon the design of a fatigue testing machine that will test simple and inexpensive specimens either in the conventional manner at a fixed amplitude or at varying stress amplitude.

The problem of obtaining sufficient data for statistical studies is always aggravated by the cost of making the large number of specimens required for statistical analysis. The authors have overcome this by designing a machine which employs straightened wire test specimens—a relatively inexpensive specimen. They have also devised a means of varying the stress amplitude in accordance with a given pattern in such a way that the change can be made without introducing extraneous stresses in the test specimen.

Miss Torrey and I are pleased that Mr. Corten has offered data which substantiate one of our suggestions, namely, that if one wishes to maintain a similar degree of confidence in tests at different stress levels, more tests should be made at the lower deflections or stress levels. Mr. Corten has offered data which definitely confirm the desirability of that suggestion.

Again, the authors should be congratulated for designing a very useful piece of equipment.

Mr. R. Ashley.[2]—Briefly, we think in terms of fatigue resistance in a textile fabric as the retained tensile strength after prolonged flexure life or the rate of tensile degradation. Normally, we think in terms of flexure life of 100 or more megacycles.

Would this type of apparatus have any use in evaluating fatigue or endurance life of twisted multi-strand wire tire cord?

Chairman Howell[3].—How was the wire straightened?

Mr. H. T. Corten (*author*).—We asked for 3-ft lengths of commercially straightened steel wire. I do know all the specimens were not straight.

In connection with multi-strand wire, I suspect this machine would not be appropriate for that purpose. The machine requires the possibility to compress the wire and multi-strand wire will probably not act as a unit when compressed.

Messrs. H. T. Corten and G. M. Sinclair (*author's closure*).—The authors wish to thank Mr. G. R. Gohn for his kind comments regarding the wire fatigue testing machine. The versatility of the machine lies in the fact that the amplitude of the alternating stress may be conveniently varied to obtain a wide variety of load histories. Well over 1000 specimens have now been tested employing various load histories. These more extensive results[4] confirm the reliability and reproducibility of the data.

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[1] Member of the Technical Staff, Bell Telephone Labs., Inc., New York, N. Y.

[2] Manager, Textile Division, General Tire and Rubber Co., Akron, Ohio.

[3] Chief, Mechanical Testing Div., Alcoa Research Labs., New Kensington, Pa.

The machine was designed and developed primarily as a research tool. To obtain the desired versatility of alternating load history, fixed length wire specimens with a limited range of diameters are required. To perform satisfactorily as a deflected rotating strut, the wire specimen must be initially straight and behave elastically when subjected to a compressive load. In view of these specimen requirements, this equipment would not be well suited for testing the twisted multi-stranded wire described by Mr. Ashley.

The wire employed in these experiments was straightened commercially by passing it between two revolving skewed rolls. Most of the 11 in. specimen lengths were found to run very true. Occasionally a specimen was encountered that did not run true and these were discarded.

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[4] H. T. Corten and T. J. Dolan, "Cumulative Fatigue Damage," session 3, paper 2, International Conference on Fatigue of Metals sponsored by the Institution of Mechanical Engineers in cooperation with American Society of Mechanical Engineers, 1956.

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON SOLDER

The problems connected with soldering are many fold, and that these problems are being attacked is evidenced by the information presented in this Symposium on Solder. These problems, which actually involve several branches of science including chemistry, physics, and metallurgy are thoroughly explored by the sixteen papers comprising this symposium. The symposium was presented at the Fifth, Ninth, Fourteenth, and Fifteenth Sessions of the Fifty-ninth Annual Meeting of the American Society for Testing Materials held in Atlantic City, N. J., June 19 and 20, 1956. It was sponsored by Committee B-2 on Non-Ferrous Metals and Alloys—with Mr. G. H. Harnden of General Electric Co., Schenectady, N. Y. serving as symposium committee chairman.

The papers presented were:

Introduction—Bruce W. Gonser

Solders

Solder, Fluxes, and Techniques for Soldering Aluminum—J. D. Dowd
Ultrasonic Soldering of Aluminum—J. Byron Jones and John G. Thomas
Dip Soldering Printed Circuits—E. S. Miller and A. A. Johns, Jr.
Soldering in Semiconductor Devices—W. F. Lootens
Industrial Survey of Paste Solder Alloys—H. R. Williams

Soldering Fluxes

Corrosive Fluxes—Their Role in Soldering—Robert M. MacIntosh
The Use of Rosin and Activated Rosin Fluxes—Frederick C. Disque, Jr.
Soldering Fluxes and Flux Principles—A. Z. Mample
Non-Corrosive Fluxes, Evaluation of Spread and Corrosion Properties—H. C. Sohl

Joint Strength Characteristics

A Method for Testing and Evaluating the Joint Properties of a Copper Liner Soldered in an Aluminum Casting—M. V. Davis
The Performance of Some Soft Solders at Elevated Temperatures and Pressures—H. E. Pattee and R. M. Evans
Dip Solder Printed Circuit Joint Characteristics—Alfred A. Johns, Jr. and E. S. Miller
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USE OF FLY ASH IN CONCRETE*

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Synopsis

The purpose of this investigation was to study the properties of concrete containing as replacement for part of the cement, four fly ashes from two widely separated areas selected because they represented sources of fly ash which have a history of extensive use in concrete. From each source fly ashes of widely different fineness were selected, with the expectation that those with the maximum fineness would have the lowest carbon content. Concrete specimens containing the fly ashes were tested for strength at various ages, for shrinkage at various periods when stored in a dry atmosphere, durability in freezing and thawing, and resistance to attack of calcium chloride used for ice removal.

These tests indicate, in general, that at ages through 28 days both the flexural and compressive strengths were lower than comparable concrete without fly ash. At one year the strengths of the concrete containing fly ash were about as high or higher than the plain concrete. Fly ash did not improve the resistance to freezing and thawing of non air-entrained concrete. In air-entrained concrete some of the fly ashes gave satisfactory durability and some did not. The particular portland cement used was also a factor. In general, concrete containing fly ash showed less shrinkage upon drying than concrete without fly ash. All substitutions of fly ash for portland cement adversely affected the resistance of both air-entrained and non air-entrained concrete to attack by calcium chloride solutions used for ice removal.

In an effort to develop a more durable concrete, research engineers have been seeking a low-cost form of finely divided silica, for addition to portland-cement concrete, which would react with the alkalies and lime liberated by the cement in the hydration process. A material which appears to have the desired properties is fly ash. This is a waste product resulting from the burning of pulverized coal and it accumulates in large quantities at the power plants. It has a high silica content and a fineness similar to cement.

Not only have claims been made for the suitability of fly ash for producing a more durable concrete, but the product has been proposed as an extender or replacement for portland cement. Impetus has been given to the use of fly ash for this purpose because of the shortage of cement during the past few years.

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TABLE I.—CHEMICAL COMPOSITION AND PHYSICAL PROPERTIES OF PORTLAND CEMENTS AND FLY ASHES.

	Portland Cement		Fly Ash			
	A	B	A	B	X	Y
Chemical composition, per cent						
Silicon dioxide	22.0	22.3	47.1	49.2	41.2	38.5
Aluminum oxide	5.6	5.4	18.2	19.9	22.1	23.5
Ferric oxide	2.5	2.4	19.2	16.2	20.6	18.8
Calcium oxide	62.8	66.1	7.0	5.5	6.0	3.2
Magnesium oxide	3.0	1.0	1.1	1.4	1.2	1.0
Sulfur trioxide	2.0	1.7	2.8	2.7	0.9	0.6
Loss on ignition	0.8	1.2	1.2[a]	1.2[a]	5.4[a]	11.6[a]
Sodium oxide	0.40	0.04	1.80	2.00	1.00	0.60
Potassium oxide	1.05	0.15	2.15	2.35	1.42	1.88
Total equiv. alkalies as Na_2O	1.09	0.14	3.21	3.55	1.93	1.84
Insoluble residue	0.16	0.12	...	...	...	...
Chloroform-soluble organic substances	0.009	0.003	...	...	...	...
Free calcium oxide	0.85	0.56	...	...	...	...
Water-soluble alkali						
Na_2O	0.11	0.01	...	...	...	...
K_2O	0.63	0.02	...	...	...	...
Computed compound composition, per cent						
Tricalcium silicate	42	55	...	...	...	...
Dicalcium silicate	31	22	...	...	...	...
Tricalcium aluminate	11	10	...	...	...	...
Tetracalcium aluminoferrite	8	7	...	...	...	...
Calcium sulfate	3.4	2.9	...	...	...	...
Carbon	...	...	0.2	0.6	5.0	11.2
Carbon dioxide	...	...	0.01	0.04	0.04	0.03
Physical properties						
Apparent specific gravity	3.20	3.17	2.49	2.52	2.51	2.43
Specific surface (Wagner) sq cm per g	1800	1625	...	...	...	...
Specific surface (Blaine) sq cm per g	...	...	3075	4305	2565	3220
Passing No. 325 sieve, per cent	...	...	93.2	95.2	84.5	89.4
Autoclave expansion, per cent	0.32	0.04	...	...	...	...
Normal consistency, per cent	25.5	25.0	...	...	...	...
Time of setting (Gillmore test)						
Initial, hr	3.2	4.2	...	...	...	...
Final, hr	5.2	6.4	...	...	...	...
Compressive strength (1:2.75 mortar)						
At 7 days, psi	2340	2960	...	...	...	...
At 28 days, psi	3670	5070	...	...	...	...
Mortar air content, per cent	9.1	6.8	...	...	...	...

[a] Determination made at 600 C.

The purpose of this investigation was to study the properties of concrete containing as replacement for part of the cement, four fly ashes from two widely separated areas selected because they represented sources of fly ash which have a history of extensive use in concrete.

SCOPE OF WORK AND MATERIALS USED

The fly ashes[2] from one area are identified hereafter as A and B, and from the other area as X and Y. From each source, fly ashes of widely different fineness were selected.

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[2] Fly ashes A, B, X, and Y are identified as Nos. 1, 3, 14, and 29, respectively, in the report by Brink and Halstead, "Studies Relating to the Testing of Fly Ash for Use in Concrete."

In this investigation specimens for compressive and flexural strength, durability and volume-change tests were made from concretes without air and with 3 to 6 per cent entrained air. Since fly ash is generally used as a replacement for part of the portland cement in paving or structural concrete, it seemed logical that the air content should be maintained within the usual limits of 3 to 6 per cent.

In order to investigate the possibility of reaction of the fly ash with the alkalies in the cement, two portland cements, one of relatively high and the other of relatively low alkali content, were used. The chemical analyses of the two portland cements shown in Table I indicate that cement A had an alkali content (1.09 per cent), higher than that permitted by AASHO specification M 85-53 for a low-alkali cement and that cement B had a very low alkali content (0.14 per cent).

Each of the four fly ashes was used as a replacement for portland cement in two percentages, $16\frac{2}{3}$ and $33\frac{1}{3}$ per cent, in the concrete for strength determinations and freezing-and-thawing and volume-change measurements. For the concrete slabs that were stored outdoors and exposed to the action of calcium chloride used for ice removal only one percentage replacement, $33\frac{1}{3}$ per cent, was used. In each case the replacement of cement by fly ash was on the basis of solid volumes.

Concrete for compressive and flexural strength test specimens was made with $1\frac{1}{2}$-in. maximum size crushed limestone, and a siliceous sand with fineness modulus of 2.75. The aggregates were of good quality and had a good service record for durability. Aggregates from the same sources were used in making the volume-change and freezing-and-thawing specimens and the outdoor exposure slabs, except that the maximum size was limited to 1 in. This maximum size was required because of the small size specimens.

MIX PROPORTIONS

The mix data for all concrete specimens are given in Table II. Mixes without fly ash were designed for a cement content of 6.0 sacks of cement per cu yd, and a slump of approximately 3.0 in. Where fly ash was used, one or two sacks of the cement per cubic yard of concrete was replaced by an equal solid volume of fly ash. Because of differences in specific gravities of the cements and the fly ashes, the weight of fly ash for a solid volume equivalent to the solid volume of a 94-lb sack of cement was 74 lb for fly ash A, B, and X, and 72 lb for fly ash Y. The quantity of sand in air-entrained mixes was reduced to compensate for the volume of added air, thus maintaining a constant cement content. For all mixes with the same maximum size coarse aggregate, the actual weight of coarse aggregate per cubic yard of concrete was constant.

The water requirement for concrete of a given slump varied with the amount and properties of the fly ash used for replacement of cement. The mix data for the strength-test specimens shown in Table II indicate that when $16\frac{2}{3}$ per cent of fly ash A or B was used, the water was approximately $1\frac{1}{4}$ gal per cu yd of concrete less than that in the mixes without fly ash; and when $33\frac{1}{3}$ per cent of fly ash A or B was used, the reduction was approximately $2\frac{1}{4}$ gal per cu yd. When either $16\frac{2}{3}$ or $33\frac{1}{3}$ per cent of fly ash X was used the water requirement was the same as for concrete without fly ash, but when $16\frac{2}{3}$ per cent of fly ash Y was used, approximately $\frac{1}{2}$ gal more

TABLE II.—CONCRETE MIX DATA.

Fly Ash			Mixes for Strength Tests[a]							Mixes for Freezing and Thawing and Volume Change Tests[b]					
Identification	Source	Amount Replacement,[c] per cent	Cement, sacks per cu yd	Fly Ash, lb per cu yd	Water, gal per cu yd	Slump, in.	Weight Plastic Concrete, lb per cu ft	Air,[d] per cent	Admixture,[e] per cent	Cement, sacks per cu yd	Fly Ash, lb per cu yd	Water, gal per cu yd	Slump, in.	Weight Plastic Concrete, lb per cu ft	Air,[d] per cent
PORTLAND CEMENT A—NON AIR-ENTRAINED CONCRETE															
None........	...	...	6.0	0	33.7	3.0	151.6	1.6	...	5.9	0	35.5	2.7	148.6	2.0
A...........	Midwest..............	16.7	5.0	74	32.9	3.1	151.6	1.5	...	5.0	74	33.0	2.5	149.1	2.8
		33.3	4.1	148	31.0	2.6	151.4	1.7	...	4.0	148	34.2	2.5	148.9	1.4
B...........	Midwest..............	16.7	5.1	74	31.8	2.6	152.0	1.5	...	5.0	74	34.8	2.4	149.4	1.3
		33.3	4.1	148	31.1	2.7	152.1	1.3	...	4.0	148	35.1	3.3	149.3	0.8
X...........	East..................	16.7	5.0	74	33.5	3.1	151.4	1.4	...	5.0	74	35.6	3.0	149.6	1.0
		33.3	4.0	148	33.7	3.2	150.9	1.2	...	4.0	148	35.0	4.0	148.4	1.5
Y...........	East..................	16.7	5.0	72	33.6	2.7	151.2	1.4	...	4.9	72	36.1	3.5	148.6	1.4
		33.3	4.0	144	34.2	3.2	150.2	1.4	...	4.0	144	35.2	3.7	147.7	1.7
PORTLAND CEMENT A—AIR-ENTRAINED CONCRETE															
None........	...	...	6.0	0	30.4	3.0	147.3	4.6	100	5.8	0	31.9	3.0	141.2	6.6
A...........	Midwest..............	16.7	5.0	74	29.4	3.0	146.6	4.9	93	4.8	74	31.4	3.5	141.0	6.2
		33.3	4.1	148	28.7	3.2	146.6	4.7	87	4.0	148	31.3	3.0	144.1	4.5
B...........	Midwest..............	16.7	5.0	74	29.2	3.0	146.4	4.8	137	5.0	74	31.0	3.0	145.0	4.2
		33.3	4.1	148	28.8	3.1	147.5	4.3	152	4.0	148	31.9	3.0	145.1	3.9
X...........	East..................	16.7	5.0	74	30.5	2.9	146.7	4.3	155	4.9	74	32.8	3.3	143.0	5.0
		33.3	4.0	148	30.8	3.2	146.2	4.2	217	4.0	148	34.8	3.0	144.4	4.0
Y...........	East..................	16.7	5.0	72	31.0	2.8	145.5	4.8	242	4.8	72	34.5	4.0	141.6	5.8
		33.3	4.0	144	32.2	3.3	144.7	4.4	395	4.0	144	36.0	3.5	144.7	3.1

PORTLAND CEMENT B—NON AIR-ENTRAINED CONCRETE

None	...	...	6.0	0	34.8	3.0	151.6	1.3	...	5.9	0	36.9	3.2	149.2	1.3
A	Midwest	16.7	5.0	74	33.3	3.1	151.4	1.4	...	4.9	74	36.9	3.0	149.3	0.7
		33.3	4.0	148	31.9	3.1	151.2	1.6	...	4.0	148	35.6	3.5	149.2	0.7
B	Midwest	16.7	5.0	74	32.8	2.9	151.6	1.5	...	4.9	74	35.8	3.5	148.9	1.3
		33.3	4.0	148	31.6	3.0	151.6	1.6	...	4.0	148	34.3	3.5	149.2	1.1
X	East	16.7	5.0	74	34.6	3.5	150.9	1.3	...	4.9	74	37.2	3.0	148.5	1.1
		33.3	4.0	148	34.5	3.5	150.5	1.2	...	4.0	148	37.0	3.2	148.4	0.8
Y	East	16.7	5.0	72	34.7	3.2	150.8	1.3	...	4.9	72	36.3	2.7	148.6	1.3
		33.3	4.0	144	35.1	2.7	149.7	1.4	...	3.9	144	36.6	3.0	147.2	1.6

PORTLAND CEMENT B—AIR-ENTRAINED CONCRETE

None	...	...	6.0	0	31.6	2.7	146.0	4.6	100	5.9	0	32.7	2.8	143.4	5.4
A	Midwest	16.7	5.0	74	30.9	3.1	146.4	4.4	89	4.9	74	32.8	3.5	144.1	4.9
		33.3	4.0	148	29.5	3.2	146.0	4.8	93	3.9	148	30.5	3.2	142.4	5.6
B	Midwest	16.7	5.0	74	30.5	3.0	146.2	4.5	143	4.9	74	31.8	3.0	143.9	4.7
		33.3	4.1	148	29.5	3.1	146.5	4.4	179	4.0	148	30.6	3.0	142.8	5.2
X	East	16.7	5.0	74	31.5	3.1	145.1	4.9	189	4.8	74	33.3	3.0	141.8	5.6
		33.3	4.0	148	31.5	3.5	144.4	4.8	261	3.9	148	34.0	4.3	140.7	5.6
Y	East	16.7	5.0	72	32.5	3.1	146.4	4.2	279	4.9	72	35.0	3.3	144.1	3.1
		33.3	4.0	144	33.1	3.7	144.8	4.3	489	3.9	144	35.1	4.0	143.7	3.7

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[a] Proportions for mixes without fly ash were as follows: For non-air-entrained concrete 94–190–350, for air-entrained concrete 94–170–350. Maximum size C.A. = 1½ in.

[b] Proportions for mixes without fly ash were as follows: For non-air-entrained concrete 94–205–330, for air-entrained concrete 94–188–330. Maximum size C.A. = 1 in.

[c] Fly ash used as a replacement for cement on a solid volume basis.

[d] Air contents for non-air-entrained concrete were calculated, whereas air contents for air-entrained concrete were determined by ASTM pressure method C 231 – 49 T.

[e] Relative amount of air entraining admixture used to produce air in concrete for strength tests. Amount used in mixes without fly ash considered as 100 per cent.

TABLE III.—RESULTS OF STRENGTH AND FREEZING-AND-THAWING TESTS.

Fly Ash		Air, per cent	Modulus of Rupture, psi			Compressive Strength, psi			Relative Strength,[a] per cent						Air, per cent	Loss in N^2,[b] per cent							Durability Factor
									Modulus of Rupture			Compressive Strength											
Identification	Amount Replacement, per cent		7 days	28 days	1 yr	7 days	28 days	1 yr	7 days	28 days	1 yr	7 days	28 days	1 yr		10 cycles	25 cycles	50 cycles	100 cycles	150 cycles	175 cycles	200 cycles	
PORTLAND CEMENT A—NON AIR-ENTRAINED CONCRETE																							
None	...	1.6	590	790	790	3160	4410	5750	100	100	100	100	100	100	2.0	25	45	(24 cycles)					6
A	16.7	1.5	525	740	835	2930	4480	6300	89	94	106	93	102	110	2.8	22	54	(24 cycles)					5
	33.3	1.7	465	720	1000	2420	4420	6390	79	92	127	77	100	111	1.4	16	56	(24 cycles)					5
B	16.7	1.5	550	790	955	2970	4640	6640	93	100	121	94	105	115	1.3	24	57	(24 cycles)					5
	33.3	1.3	485	855	1030	2510	4640	6140	82	108	130	79	105	107	0.8	16	48	(24 cycles)					6
X	16.7	1.4	525	770	830	2620	4220	5960	89	97	105	83	96	104	1.0	31	44	(14 cycles)					4
	33.3	1.2	360	665	880	1900	3350	5770	61	84	111	60	76	100	1.5	41		(7 cycles)					2
Y	16.7	1.4	515	640	865	2450	3900	6020	87	81	109	78	88	105	1.4	38		(7 cycles)					2
	33.3	1.4	355	610	835	1690	3270	4970	60	77	106	53	74	86	1.7	51		(6 cycles)					1
PORTLAND CEMENT A—AIR-ENTRAINED CONCRETE																							
None	...	4.6	595	790	745	3100	4140	5410	100	100	100	100	100	100	6.6	5	4	9	9	14	16	18	82
A	16.7	4.9	530	715	790	2670	3970	5620	89	91	106	86	96	104	6.2	4	3	13	7	12	15	20	80
	33.3	4.7	445	740	825	2240	3780	5750	75	94	111	72	91	106	4.5	5	4	15	12	24	28	34	66
B	16.7	4.8	505	785	860	2720	4040	5970	85	99	115	88	98	110	4.2	6	4	14	17	24	32	37	63
	33.3	4.3	445	785	930	2410	4030	6000	75	99	125	78	97	111	3.9	5	5	6	10	23	31	39	61
X	16.7	4.3	515	770	830	2530	3660	5580	87	97	111	82	88	103	5.0	7	6	7	13	23	28	29	71
	33.3	4.2	355	640	865	1810	3260	5240	60	81	116	58	79	97	4.0	8	7	12	23	48	(150 cycles)		40
Y	16.7	4.8	470	660	760	2230	3490	5410	79	84	102	72	84	100	5.8	5	5	6	6	14	18	19	81
	33.3	4.4	335	555	760	1490	2640	4400	56	70	102	48	64	81	3.1	10	11	20	39	(86 cycles)			25

PORTLAND CEMENT B—NON AIR-ENTRAINED CONCRETE

None	...	1.3	705	880	850	3770	5890	7480	100	100	100	100	100	100	1.3	4	9	42			(52 cycles)		15
A	16.7	1.4	640	790	935	3150	5220	8690	91	90	110	84	89	116	0.7	3	8	25	56		(85 cycles)		19
	33.3	1.6	535	815	1025	2380	4460	8250	76	93	121	63	76	110	0.7	5	15	38			(52 cycles)		16
B	16.7	1.5	575	915	1035	3160	5580	8730	82	104	122	84	95	117	1.3	2	12	46			(48 cycles)		14
	33.3	1.6	530	905	1100	2740	4970	9020	75	103	129	73	84	121	1.1	4	26	47			(34 cycles)		9
X	16.7	1.3	555	750	915	2620	4490	8050	79	85	108	69	76	108	1.1	2	12	46			(48 cycles)		14
	33.3	1.2	460	645	935	1820	2960	6740	65	73	110	48	50	90	0.8	9	36	44			(34 cycles)		9
Y	16.7	1.3	620	770	910	2660	4390	7730	88	88	107	71	75	103	1.3	4	14	63			(48 cycles)		11
	33.3	1.4	425	690	995	1750	3130	6570	60	78	117	46	53	88	1.6	36	51				(14 cycles)		3

PORTLAND CEMENT B—AIR-ENTRAINED CONCRETE

None	...	4.6	700	775	750	3840	5480	6270	100	100	100	100	100	100	5.4	2	4	4	4	9	8	9	91
A	16.7	4.4	650	730	860	3320	4800	7260	93	94	115	86	88	116	4.9	1	3	5	1	9	7	14	86
	33.3	4.8	495	750	900	2590	4200	7110	71	97	120	67	77	113	5.6	0	3	1	2	8	7	7	93
B	16.7	4.5	600	750	875	3450	5290	7540	86	97	117	90	97	120	4.7	0	5	1	5	6	11	14	86
	33.3	4.4	570	805	875	2850	4510	7440	81	104	117	74	82	119	5.2	0	7	2	7	7	8	9	91
X	16.7	4.9	575	735	825	2860	4490	6710	82	95	110	74	82	107	5.6	0	4	2	8	6	7	9	91
	33.3	4.8	445	635	835	1970	3310	5700	64	82	111	51	60	91	5.6	0	2	4	6	11	14	16	84
Y	16.7	4.2	555	745	825	2870	4400	6500	79	96	110	75	80	104	3.1	0	4	6	5	6	8	8	92
	33.3	4.3	370	605	780	1840	2870	5420	53	78	104	48	52	86	3.7	0	4	8	7	15	16	16	84

Each value for mixes without fly ash is an average of six tests; for mixes containing fly ash each value is an average of three tests.

[a] Based on strength of concrete without fly ash.

[b] Figures in parentheses indicate number of cycles at which final reading was taken.

water was required, and when $33\frac{1}{3}$ per cent of fly ash Y was used approximately 1 gal more water was required. The same trends were noted for the concrete used in the freezing-and-thawing and volume-change tests.

The quantity of air-entraining admixture required for a constant air content increased with increases in the carbon content of the fly ash and with the quantity of fly ash used as a replacement for the portland cement. This variation is shown in Table II. When either $16\frac{2}{3}$ or $33\frac{1}{3}$ per cent of fly ash A was used, approximately 10 per cent less air-entraining agent was required than was needed for the concrete with 100 per cent portland cement, and when $33\frac{1}{3}$ per cent fly ash Y was used with cement B, approximately five times as much air-entraining admixture was required.

Slump and air content were maintained approximately constant for all concretes.

MIXING AND FABRICATION OF SPECIMENS

All mixing was done in an open-pan type mixer of $1\frac{3}{4}$ cu ft capacity. The following mixing cycle was employed: Cement plus the fly ash and damp sand were mixed for 30 sec, after which the estimated total quantity of mixing water plus the air-entraining admixture, if any, was added and the mortar mixed for a further period of 1 min. Coarse aggregate, in a saturated surface-dry condition, was then added and the concrete mixed for an additional 2 min, making a total mixing time of $3\frac{1}{2}$ min. Air contents for the air-entrained concrete were determined by the pressure method (ASTM Method C 231 - 49 T),[3] and for the non air-entrained concrete, air content was calculated by the gravimetric method (ASTM C 138 - 44).[3] Consistency was controlled by means of the slump test.

TESTING PROCEDURES

Strength specimens were molded, cured, and tested in accordance with applicable ASTM procedures. All strength specimens were moist cured continuously until tested.

The freezing-and-thawing tests were made on prisms 3 by 4 by 16 in. sawed from 4 by 16 by 24-in. slabs. The concrete slabs were cured in the moist room for 14 days, then stored in laboratory air for 7 days at the end of which time seven 3 by 4 by 16-in. prisms were sawed from each slab. Three were used for the freezing-and-thawing test and the other four were held in moist air storage for future tests. The freezing-and-thawing prisms were returned to the moist room for 7 days, then immersed in water for 7 days, and at a total age of 35 days the freezing-and-thawing cycle was started.

A 24-hr cycle was used in making the freezing-and-thawing tests. Each specimen was frozen and thawed in water in an individual metal container of such size as to provide about $\frac{1}{8}$-in. clearance on all sides. The specimens in the containers were placed vertically in the freezing chamber. The refrigerant used was alcohol, and it entirely surrounded the sides of the container to a height of about 18 in. The specimens in the containers were frozen at −10 F for 18 hr, then removed from the freezer and thawed in a water bath at 70 F for 6 hr. After each cycle, the specimens were removed from the containers, turned upside down, and replaced. The water in the containers was changed.

Resistance to the effects of alternate freezing and thawing was determined periodically during the test by measuring changes in the dynamic modulus of elasticity of the concrete. For a given specimen, changes in dynamic modulus of elasticity, *E*, are proportional to changes in the square of the natural frequency of vibration, N^2. It is, therefore, possible to use the reduction in N^2 directly as a measure of the deterioration of the concrete. Freezing and thawing was continued until the specimens showed a loss in N^2 of 40 per cent or for 200 cycles, whichever occurred first.

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[3] 1955 Book of ASTM Standards, Part 3.

In addition to the 4 by 16 by 24-in. slabs which were sawed for the freezing-and-thawing prisms, six 3 by 4 by 16-in. beams were made for volume change tests. These specimens were removed from the molds after 24 hr under moist burlap and then were stored in laboratory air at 72 F and 50 per cent relative humidity. After 180 days in laboratory air they were stored in water for 14 days and then returned to laboratory air for an additional 180 days. The length of each specimen was determined when it was taken out of the mold and periodically thereafter.

Additional 4 by 16 by 24-in. slabs were made later to determine the effect of fly ash on the resistance of concrete to scaling due to freezing and ice removal with calcium chloride. A raised edge was cast around the perimeter of each slab. In these tests only concretes containing 33⅓ per cent replacement of each of the four fly ashes for both cements were used with the non air-entrained and the air-entrained concrete. The slabs were cast in June 1952 and were stored in moist air for 30 days. They were then placed outdoors on the ground. During the fall and winter season the top surface of each slab was kept covered with water. When ice was frozen on the slabs, calcium chloride was applied to the surface at the rate of 2.4 lb per sq yd. After the ice was completely thawed, the surface was washed and fresh water left on the surface to await another freezing. During the winter of 1952–53, 17 cycles of freezing and thawing were obtained, during the winter of 1953–54, 34 cycles, and during the winter of 1954–55, 29 cycles, for a total of 80 cycles. The slabs were rated periodically for surface scale.

TABLE IV.—VISUAL RATINGS OF SURFACE SCALING OF CONCRETE SLABS EXPOSED TO ACTION OF CALCIUM CHLORIDE.

Fly Ash		Air,[a] per cent	Rating After Exposure to Outdoor Freezing and Thawing[b] with $CaCl_2$ for Cycles Indicated				
Identification	Replacement per cent		12	17	39	51	80
PORTLAND CEMENT A—NON AIR-ENTRAINED CONCRETE							
None	...	1	5	8	10	10	10
A	33.3	1	6	8	10	10	10
B	33.3	1	6	8	10	10	10
X	33.3	1	7	8	10	10	10
Y	33.3	1	6	8	10	10	10
PORTLAND CEMENT A—AIR-ENTRAINED CONCRETE							
None	...	4.0	1	2	2	3	4
A	33.3	4.6	2	4	3	4	5
B	33.3	5.4	3	4	4	5	7
X	33.3	4.9	4	6	7	8	8
Y	33.3	4.3	4	6	6	8	8
PORTLAND CEMENT B—NON AIR-ENTRAINED CONCRETE							
None	...	1	3	8	10	10	10
A	33.3	1	4	8	9	10	10
B	33.3	1	4	8	10	10	10
X	33.3	1	6	8	10	10	10
Y	33.3	1	5	8	10	10	10
PORTLAND CEMENT B—AIR-ENTRAINED CONCRETE							
None	...	6.8	1	2	2	2	3
A	33.3	4.0	1	2	4	4	5
B	33.3	3.3	5	6	7	8	8
X	33.3	3.9	6	8	8	8	8
Y	33.3	5.8	6	8	8	8	9

[a] Mix same as used for freezing-and-thawing specimens except for air content.

[b] Specimens were 4 by 16 by 24-in. slabs.

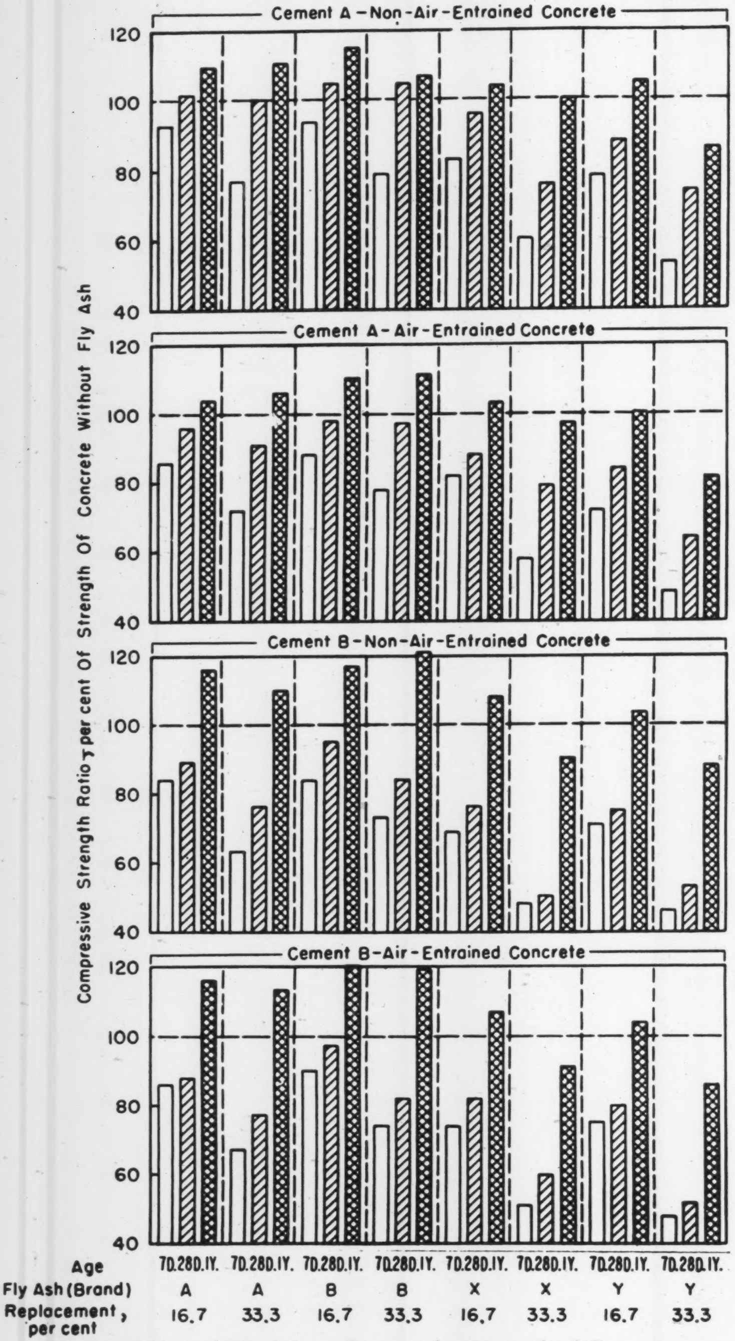

FIG. 1.—Effect of Fly Ash on Compressive Strength of Concrete.

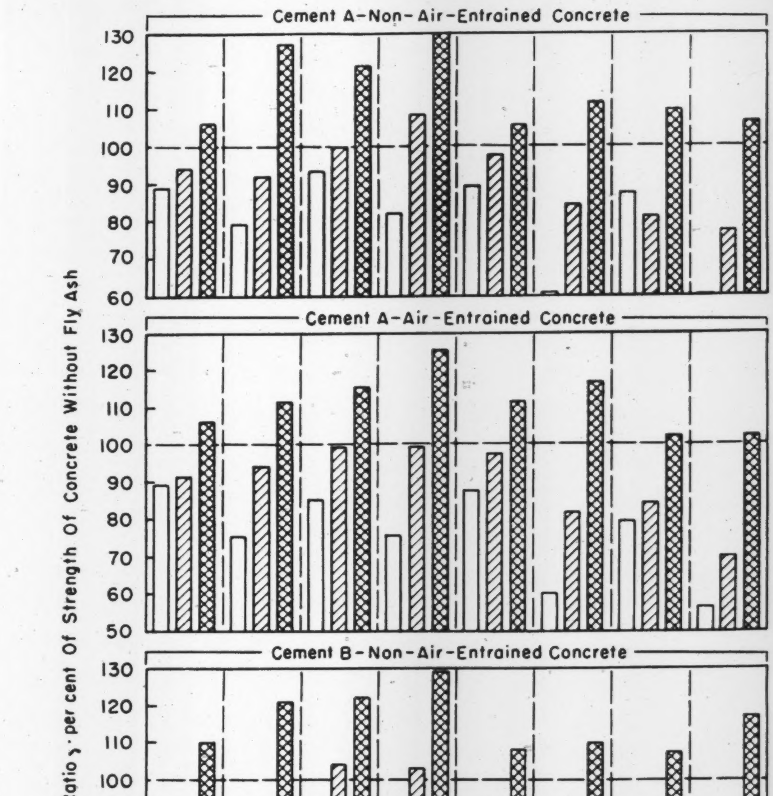

FIG. 2—Effect of Fly Ash on Flexural Strength of Concrete.

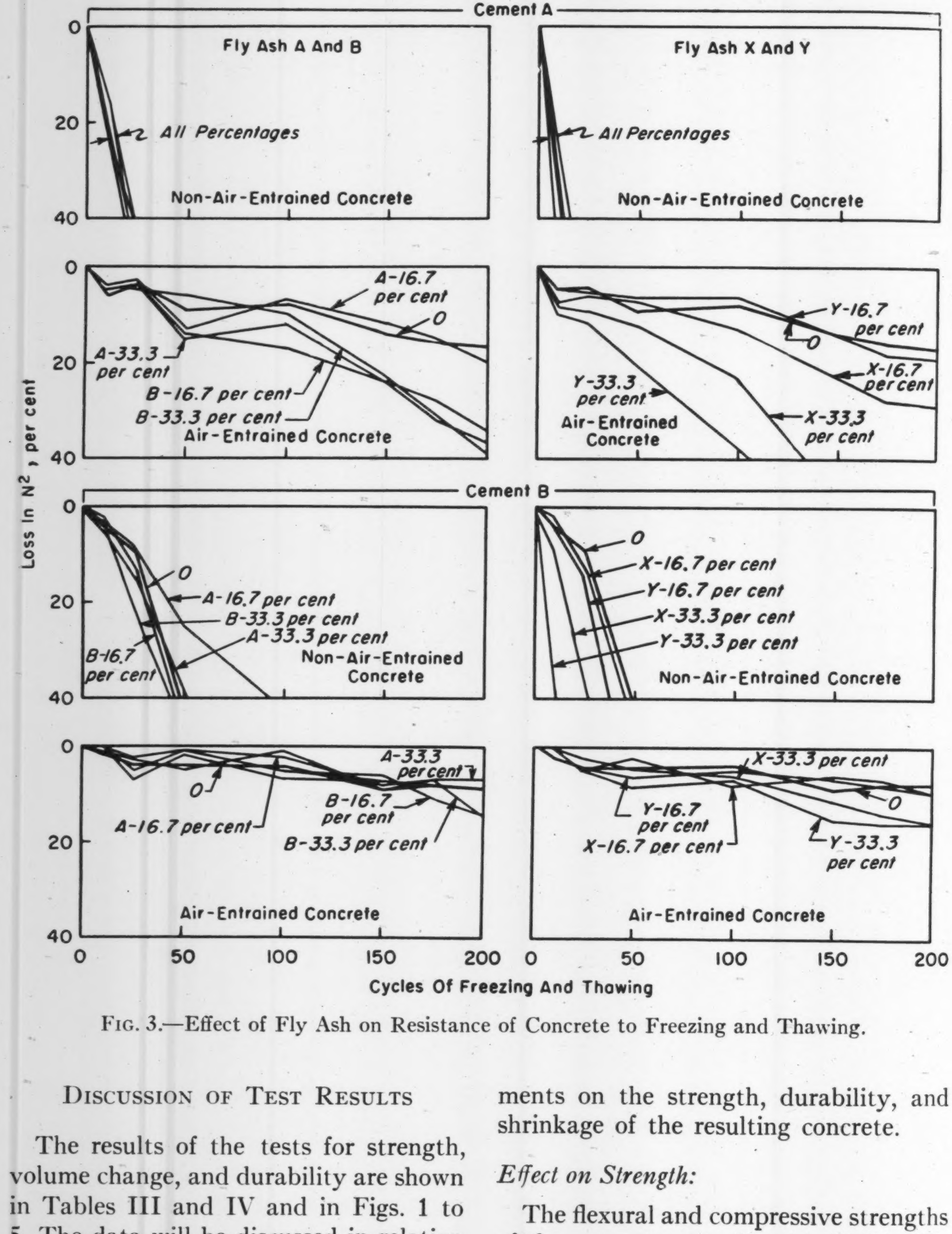

FIG. 3.—Effect of Fly Ash on Resistance of Concrete to Freezing and Thawing.

DISCUSSION OF TEST RESULTS

The results of the tests for strength, volume change, and durability are shown in Tables III and IV and in Figs. 1 to 5. The data will be discussed in relation to the effect of substituting in two percentages each of the four fly ashes for a portion of each of the two portland cements on the strength, durability, and shrinkage of the resulting concrete.

Effect on Strength:

The flexural and compressive strengths of the concretes are shown in Table III. Also shown are the relative strengths of concretes containing fly ash expressed as percentages of the strengths developed

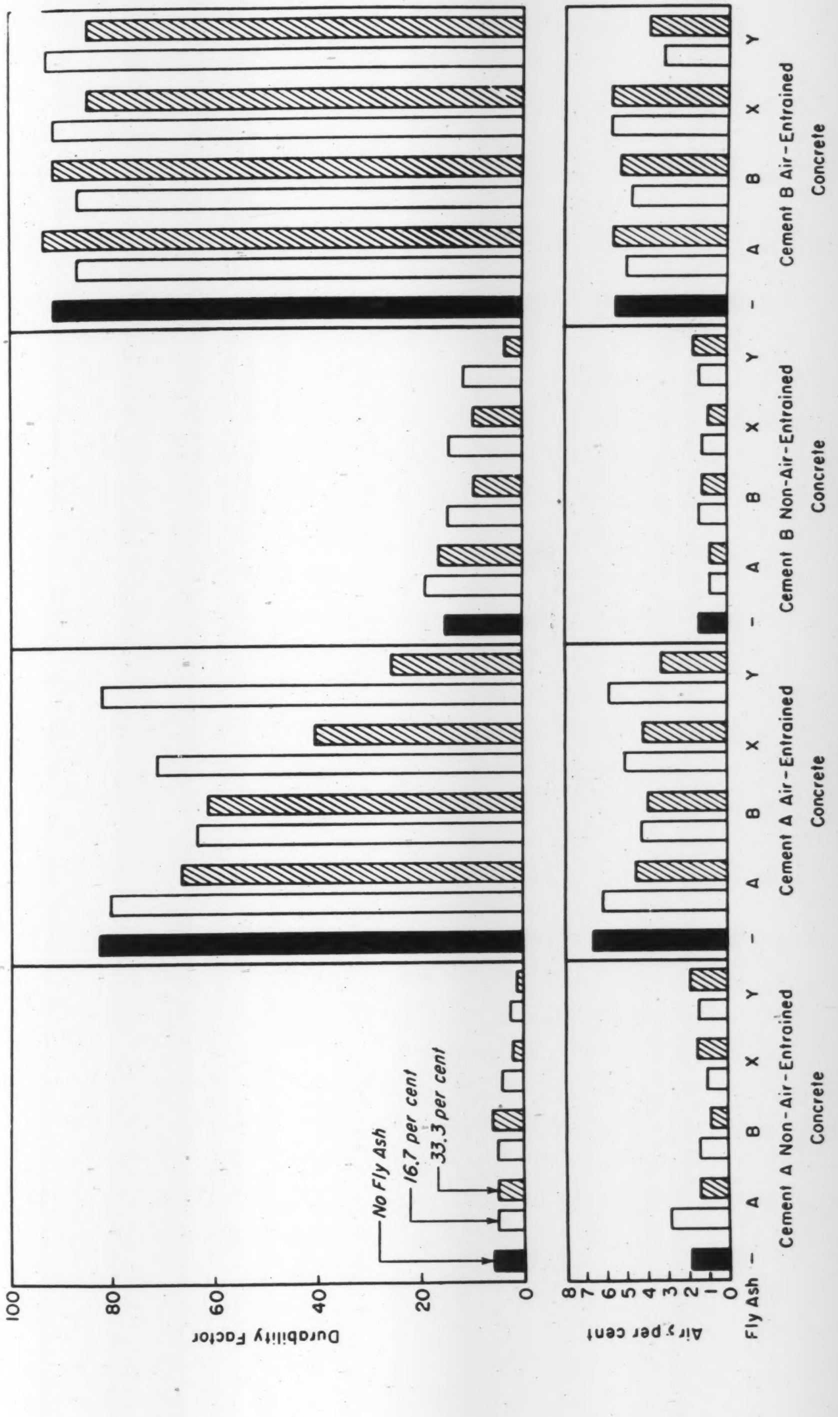

Fig. 4—Effect of Fly Ash on Durability of Concrete.

by the concrete without fly ash. These relative strengths for various ages of test are shown graphically in Figs. 1 and 2.

In the upper diagram of Fig. 1 are shown the relative compressive strengths of the non-air-entrained concretes made with cement A containing the substitutions of the two percentages of each of the four fly ashes. The 7-day strengths of concretes containing fly ash ranged from 53 to 94 per cent of the strength of comparable concrete without fly ash. At 28 days the concretes containing fly ash A or B were equal to or greater than the 28-day strength of the concrete without fly ash. The concretes containing fly ash X or Y at 28 days ranged in relative strength from 74 to 96 per cent.

All of the non-air-entrained concrete with cement A containing fly ash equalled or exceeded the strength of comparable concrete without fly ash at 1 yr with the exception of that containing $33\frac{1}{3}$ per cent of fly ash Y. The concrete containing $33\frac{1}{3}$ per cent fly ash Y had a relative strength of 86 per cent of the comparable concrete without fly ash. The highest relative strength at 1 yr for concrete made with cement A was 115 per cent.

The three remaining diagrams in Fig. 1 show a pronounced similarity in the relative strength to that shown in the first diagram. This is also apparent in the following tabulation showing the range and averages of relative strengths:

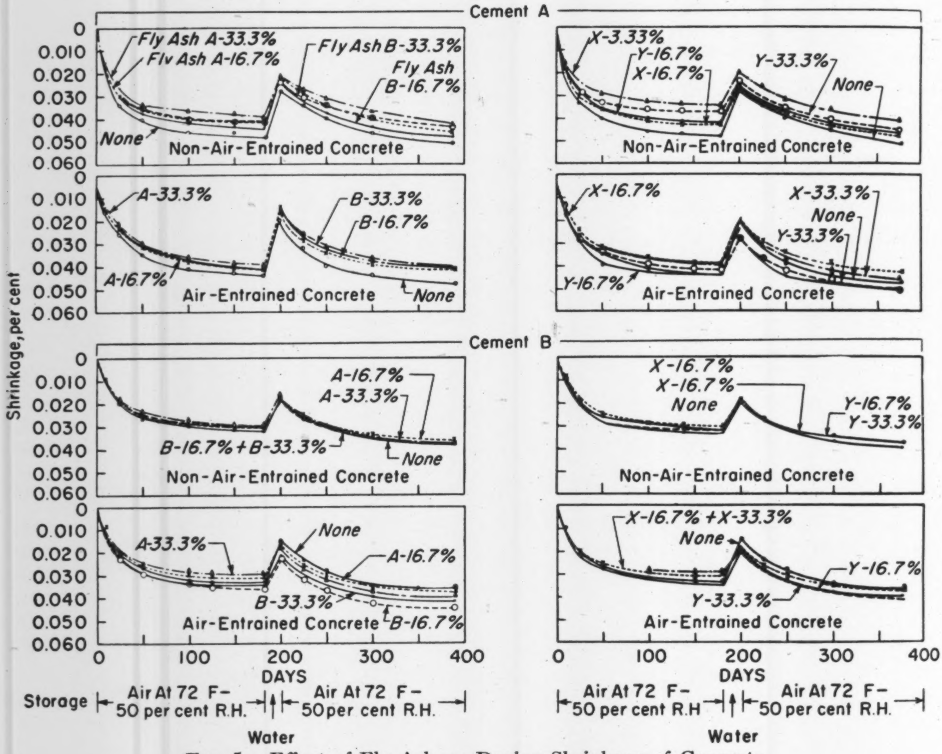

FIG. 5.—Effect of Fly Ash on Drying Shrinkage of Concrete.

	Fly Ash, per cent	Relative Compressive Strengths					
		7 days		28 days		1 yr	
		Range	Avg.	Range	Avg.	Range	Avg.
Cement A—non-air entrained	16.7	94 to 78	87	105 to 88	98	115 to 104	108
	33.3	79 to 53	67	105 to 74	89	111 to 86	101
Cement A—air-entrained	16.7	88 to 72	82	98 to 84	92	110 to 100	104
	33.3	78 to 48	64	97 to 64	83	111 to 81	99
Cement B—non-air-entrained	16.7	84 to 69	77	95 to 75	84	117 to 103	111
	33.3	73 to 46	58	84 to 50	66	121 to 88	102
Cement B—air-entrained	16.7	90 to 74	81	97 to 80	87	120 to 104	112
	33.3	74 to 48	60	82 to 52	68	119 to 86	102

It is significant that the 7-day compressive strengths of the concretes containing fly ash are in each case lower than those of the comparable concrete without fly ash. With the exception of cement A, non-air-entrained concrete containing fly ash A or B, the same relation is found at 28 days. For the fly ashes of low carbon content, both replacements of cement with fly ash resulted in concrete strengths at 1 yr greater than those of the concrete without fly ash, in one case by as much as 21 per cent. Replacement of $16\frac{2}{3}$ per cent of the cement with fly ashes X and Y resulted in somewhat lower relative strengths at 1 yr, but in all cases these were equal to or greater than 100 per cent. Concretes containing the $33\frac{1}{3}$ per cent of high-carbon fly ashes (X or Y) at all ages including 1 yr had strengths less than the concrete without fly ash except fly ash X which had approximately the same strength at 1 yr.

The use of air entrainment appeared to have little effect on the relative strengths, particularly with cement B. For each combination of cement and fly ash, the concrete with and without air entrainment had similar relative compressive strengths.

TABLE V.

	Fly Ash, per cent	Relative Flexural Strengths					
		7 days		28 days		1 yr	
		Range	Avg.	Range	Avg.	Range	Avg.
Cement A—non air-entrained	16.7	93 to 87	90	100 to 81	93	121 to 105	110
	33.3	82 to 60	70	108 to 77	90	130 to 106	118
Cement A—air-entrained	16.7	89 to 79	85	99 to 84	93	115 to 102	108
	33.3	75 to 56	66	99 to 70	86	125 to 102	113
Cement B—non-air-entrained	16.7	91 to 79	85	104 to 85	92	122 to 107	112
	33.3	76 to 60	69	103 to 73	87	129 to 110	119
Cement B—air-entrained	16.7	93 to 79	85	97 to 94	96	117 to 110	113
	33.3	81 to 53	67	104 to 78	90	120 to 104	113

Figure 2 shows a bar graph for the relative flexural strengths. The same general relation shown for compressive strengths exists for the flexural strengths, as is apparent in Table V tabulation, except that in no case did the concrete with the fly ash substitutions fail to exceed the flexural strength of the concrete without fly ash at the 1 yr period.

Durability:

The data from the freezing-and-thawing tests are shown in Table III and in Figs. 3 and 4. In Table III are shown the losses in natural frequency squared, N^2, at 10, 25, 50, 100, 150, 175, and 200 cycles and the durability factor.

The reductions in N^2 plotted against the number of cycles in Fig. 3 show that all the concretes containing fly ash and without air entrainment deteriorated very rapidly under freezing and thawing. With two exceptions all air-entrained concrete with and without fly ash substitutions withstood 200 cycles, with a reduction in N^2 of less than 40 per cent. The exceptions were the concretes made from cement A containing $33\frac{1}{3}$ per cent of fly ash X and Y which had air contents of 4.0 and 3.1 per cent, respectively.

In order to express in a single factor the loss in N^2 as related to the number of cycles of freezing and thawing required to produce that loss, a value known as the durability factor (DF)[4] is used. The values for durability factor shown in Table III are calculated as follows:

$$\mathrm{DF} = \frac{PN}{M}$$

where:

DF = durability factor of the test specimens,

P = relative dynamic modulus of elasticity at N cycles, per cent,

N = number of cycles at which P reached 60 per cent, or 200, whichever is less, and

M = 200

The durability factors and the air contents of the concrete are shown graphically in Fig. 4.

To insure satisfactory resistance of the concrete to the alternate freezing and thawing in the type of freezing test used, the durability factor should be 50 or greater. Durability factors for the concretes without entrained air were all less than 20. All the concretes without air made with cement A had durability factors of 8 or less.

Of the 18 concretes in which air was entrained only two had durability factors of less than 60. The two exceptions contained portland cement A, with $33\frac{1}{3}$ per cent of fly ash X, and with $33\frac{1}{3}$ per cent of fly ash Y, both high-carbon fly ashes. The comparable concretes made with cement B had durability factors of over 80. For air-entrained concrete containing cement A with fly ash substitutions there appears to be a general relation between air content and durability factor, the higher air contents being associated with greater durability. This relation was not apparent for cement B. (It should be pointed out that it was very difficult to obtain a uniform air content because concretes with the high-carbon fly ashes required such large amounts of the air-entraining admixture to give the desired air content.) Although the air content of the concrete in all cases was raised above 3 per cent by the use of excessive quantities of the air-entraining admixture, the concrete containing fly ash X used as a replacement for $33\frac{1}{3}$ per cent of cement A with an air content of 4.0 per cent had a durability factor of 40 and that containing fly ash Y with 3.1 per cent of entrained air, a durability factor of 25. In the concretes in which $16\frac{2}{3}$ per cent of cement A was replaced with fly ashes X and Y the air contents were 5.0 and 5.8 per cent and the durability factors 71 and 81, respectively.

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[4] See Method of Test for Resistance of Concrete to Slow Freezing and Thawing in Water or Brine, (C 292-52 T), 1955 Book of ASTM Standards, Part 3.

The durability factor for all air-entrained concretes made with cement B were 84 or above even though the concrete with the $16\frac{2}{3}$ per cent of fly ash Y had an air content of only 3.1 per cent.

The air-entrained concrete containing cement A without fly ash had a durability factor of 82, and with cement B, a factor of 91. These high factors indicate very little difference in durability between the two cements and offer no explanation as to why they act differently in concrete when fly ash is substituted for part of the cement.

Volume Change:

The volume change data for all the concretes are shown graphically in Fig. 5. The values plotted in the diagrams were actual test values. These curves show that there is very little difference in the volume change characteristics of the concrete made without fly ash substitutions and those containing various percentages of fly ash. Also, those containing the higher percentages of fly ash in general had less shrinkage than those with the smaller percentage of fly ash.

Resistance to Scaling Due to the Use of $CaCl_2$ as a Thawing Agent:[5]

Calcium chloride, frequently used for thawing ice on concrete pavements, has been found to be very injurious to non-air-entrained concrete. Concrete containing purposely entrained air usually offers good resistance to attack from chloride salts.

Table IV shows periodic visual ratings of exposure slabs at 12, 17, 39, 51, and 80 cycles. The following tabulation describes the numerical significance of the ratings:

Rating of 0—No scaling.
1—Very light spotted scale.
2—Spotted scale.
3—Light scale over about one-half the surface.
4—Light scale over most of surface.
5—Light scale over most of surface, few moderately deep spots.
6—Moderately deep scale spotted.
7—Moderately deep scale over one-half the surface.
8—Moderately deep scale over entire surface.
9—Deep scale spotted, otherwise moderate scale.
10—Deep scale over entire surface.

The concretes without air entrainment and without fly ash had very poor resistance to attack by the chloride salt. None of the fly ashes used as replacements for part of the cement were effectual in improving the resistance of non-air-entrained concrete to attack by calcium chloride.

Entrained air greatly increased the resistance of the concrete without fly ash. In air-entrained concrete all the fly ash replacements for cement were detrimental to the resistance to scaling of the concrete, as indicated by the ratings of 3 to 8 at 39 cycles as compared to 2 for the concretes without fly ash. The extent of attack by calcium chloride did not appear to differ much with the brand of cement or with the fly ash used as a replacement.

It would appear from these data that if there is a possibility that calcium chloride might be used for ice removal the use of fly ash as a substitute for portland cement in the concrete in the percentage used in this investigation is questionable.

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[5] Data up to 51 cycles reported in Highway Research Board Bulletin 125, "Resistance of Concrete Surfaces to Scaling Action of Ice-Removal Agent." Also published in *Public Roads*, Vol. 28, No. 7, April 1955.

CONCLUSIONS

An important indication of these tests where fly ash was used as a replacement for part of the cement was the importance of the carbon content of the fly ash. In general, the higher the amount of carbon in the fly ash and the greater the percentage of fly ash used as replacement of cement, the lower was the compresive and flexural strength at all ages.

The detailed conclusions are as follows:

1. The 7-day compressive strengths of concretes containing fly ash are lower than comparable concrete without fly ash. With a few exceptions, the same relation was found for the 28-day strengths. The lower strengths were always obtained with the concretes containing the fly ashes of highest carbon content. At 1 yr the strength of the concrete containing fly ash of low carbon content was always higher than that of the concrete without fly ash, in some cases by as much as 20 per cent, and the strength of the concretes containing 16⅔ per cent of fly ashes X and Y equalled or exceeded those without fly ash. Only the concretes containing 33⅓ per cent of fly ashes X and Y (high carbon content) failed to meet or surpass the strengths of the control concrete at 1 yr.

2. Much the same general relations hold for flexural strength as were obtained for compressive strength except that at 1 yr the flexural strength of all concrete with fly ash replacement was greater than that of concrete without fly ash.

3. Non-air-entrained concrete with and without fly ash had poor resistance to freezing and thawing.

4. Satisfactory durability was obtained with all air-entrained concretes containing cement A, the high-alkali cement, and the 16⅔ per cent replacement of the four fly ashes and the 33⅓ per cent replacement of fly ash A and B, but the concrete with 33⅓ per cent replacement of cement A with fly ashes X and Y (the high-carbon fly ashes) had poor durability as indicated by durability factors of 40 or lower.

5. The effect on durability and strength of concrete containing fly ash from a given source of supply varies with the characteristics of the cement used. Therefore, for cement-fly ash combinations for which no service record is available, laboratory investigations should be made prior to use.

6. In general the concrete containing fly ash showed less shrinkage upon drying than the concrete without fly ash.

7. Non-air-entrained concrete with and without fly ash was severely attacked by calcium chloride used for ice removal. The two air-entrained concretes without fly ash had good resistance to attack by calcium chloride, but all substitutions of fly ash for portland cement lowered the resistance to scaling.

8. The amount of air-entraining material necessary to produce a given amount of air in concrete increases with the carbon content of the fly ash.

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DISCUSSION

MR. C. E. LOVEWELL[1] (*by letter*).—These tests made in conjunction with those by Brink and Halstead involved the use of two low-carbon fly ashes and two higher carbon fly ashes with a low alkali and a high alkali portland cement, respectively, in concrete mixtures.

At this point we should review certain well-known facts about fly ash. McMillan and Powers demonstrated in their ACI paper in 1937[2] that any percentage replacement of portland cement by fly ash on an absolute volume basis resulted in lower strengths up to about 28 days but in greater strengths at 6 months and beyond when the percentage was 30 or below. In some types of work, such as mass concrete, this pattern of strength gain is perfectly satisfactory. However, paving and structural concrete frequently is specified with a 14-day minimum strength requirement. With this in mind, in the Chicago area, mixes are redesigned, using as much as twice the absolute volume of fly ash for cement removed but limiting the removal of cement to not more than one bag per cubic yard. Sand to total aggregate ratios are reduced according to accepted mix design theory and mixing water generally reduced to give the same slump as the comparable straight portland mix. By this redesign method, early strengths, even 1, 2, or 3-day strengths, are approximately the same as comparable straight portland-cement mixes.

Hence, the conclusion by the authors that, in general, strengths of fly ash mixes up to 28 days were lower in the $16\frac{2}{3}$ per cent and $33\frac{1}{3}$ per cent absolute volume fly ash replacement mixes is not surprising.

Regarding durability, the fly ash concretes, particularly on the $16\frac{2}{3}$ per cent replacement basis, showed durability factors practically the same (see Fig 4) as those with straight portland cement (some higher, some lower).

The summary statement that "All substitutions of fly ash for portland cement adversely affected the resistance of both air-entrained and non-air-entrained concrete to attack by calcium chloride solutions" should be read with several important reservations in mind as revealed by study of Table IV. In the first place, only the $33\frac{1}{3}$ per cent replacement was used in making this particular test. This means that concrete containing six bags of portland cement was compared with concrete containing four bags of portland cement plus two bags (74 lb each) of fly ash. Table IV reveals that the 28-day strength of this fly ash concrete was 66, 68, 83 and 89 per cent, respectively, of the respective control mixes. Although strength is not always a criterion of durability, it is reasonable to assume that a redesigned fly ash concrete mix with equal strength and equal air-entrainment to that of the straight portland-cement mix would have had better resistance to both freezing and thawing and calcium chloride salts than the fly ash mixes reported in the paper. No fly ash producer would recommend so great a replacement of portland cement by fly ash for highway paving, where early strength is so important. For instance in AIA File 3-B-2 "Chicago Fly Ash in Quality Concrete Mixes",[3] reduction of cement from 6 bags to 5¼ bags and the addition of 100 lb (1⅓ bags) of fly ash, with a reduction of 2 per cent in sand-to-total-aggregate ratio, is recommended.

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[1] Sales Manager and Service Engineer, Chicago Fly Ash Co., Chicago, Ill.

[2] F. R. McMillan and T. C. Powers, Portland Cement Assn. Research Laboratories, *Journal*, American Concrete Institute, Vol. 34, pp. 129–143 (1937).

Some tests currently are being run on an accelerated basis, comparing the straight six-bag mix with the adjusted fly ash mix as above recommended, both properly air-entrained, to determine relative resistance to scaling attack by calcium chloride solutions under freezing-and-thawing conditions.

Adding to the information in these papers the wealth of experience gained in designing several million cubic yards of fly ash concrete of all kinds, the writer would add the following conclusions:

1. It is now possible to specify fly ash for concrete for paving and structures with assurance of satisfactory results.

2. Provided concrete mix designs are used where the reduction in portland cement per cubic yard is limited to not more than one bag and the designs are based on compensating for reduction of portland cement by use of enough fly ash to give approximately equal 28-day strengths as equivalent straight portland mixes, the other important properties of the fly ash mixes will be at least as good as the straight portland-cement mixes with which they are compared. This assumes equal air content, where air-entrained concrete is called for. Likewise, it assumes reduction in sand-to-total aggregate ratio where the cement plus fly ash volume in fly ash mixes is greater than the volume of cement alone in the straight portland cement mixes. Also it assumes adjustment of water content to give equal slump of concrete to be tested.

None of our comments should be considered as criticism of the work at the Bureau of Public Roads. Uniformity of test procedures in the interest of reducing variables to a minimum probably prompted the absolute volume replacement method. Also, it should be remembered, early in 1951 when this test program was set up, this mix design method to achieve comparable early strengths had not been worked out. Desire to blanket the range of probable use no doubt led to the selection of the 16⅔ per cent and the 33⅓ per cent replacements, respectively. Space and personnel limitations could easily have prompted limiting the scaling tests to just one of the percentages of replacement, and the selection of the 33⅓ per cent replacement for this test was probably prompted by a desire to learn the worst possible condition that might occur. Note in this connection the following quote, "It would appear from these data that if there is a possibility that calcium chloride might be used for ice removal, the use of fly ash as a substitute for portland cement in the concrete in the percentage used in this investigation is questionable." As previously stated, we would not recommend a 33⅓ per cent substitution for paving concrete, but we regard these data as valuable evidence to confirm this conviction.

We would be remiss in our desire to be factual if we did not point out that even the 33⅓ per cent fly ash mixes tested as much as 19 per cent higher than the straight portland-cement mixes in flexural strength at 1 yr. This should be of real interest to the highway engineer who hopes that the pavements he designs will remain crack-free for many years.

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[3] "Use of Chicago Fly Ash in Modern Concrete Mixes," *AIA File 3-B-2*, published by the Chicago Fly Ash Co., Chicago, Ill.

Utilization of the data presented cannot help but further improve the quality of fly ash concrete in the future.

MESSRS. A. G. TIMMS AND W. E. GRIEB (*author's closure*).—The Bureau of Public Roads is now making a limited series of supplementary tests of concrete containing several of the fly ashes which were used in the original tests. Among the variables being studied is the effect of using fly ash as a replacement for cement as recommended in the discussion by Mr. Lovewell. The concrete was redesigned by the addition of 100 lb of fly ash with a compensating reduction of both cement and sand. The cement was reduced from 6 bags to 5¼ bags, and the sand-to-total aggregate ratio was reduced 2 per cent.

Flexural and compressive strength tests to an age of only 7 days have been completed. These show that the strength of the concrete containing fly ash is slightly less than the strength of corresponding concrete not containing fly ash. This finding agrees with the results of tests made by other laboratories. Specimens for tests of the resistance of the redesigned concrete to scaling by calcium chloride used for the removal of ice are being prepared and will be subjected to outdoor exposure.

In quoting relative strengths at an age of 28 days of concrete containing fly ash, Mr. Lovewell gave values for compressive strength only. The values for flexural strength are 87, 90, 86, and 90 percent, which values are somewhat higher than those for compressive strength.

STUDIES RELATING TO THE TESTING OF FLY ASH FOR USE IN CONCRETE*

By R. H. Brink[1] and W. J. Halstead[2]

Synopsis

Recent interest in the use of fly ash in concrete for highways has created a need for suitable specifications and methods of test for this material when it is used to replace part of the cement. The effect of fly ash on the strength of portland cement-fly ash mortars is considered to indicate the probable effect on the strength of concrete and is used in this investigation as a criterion for evaluating other tests of fly ash. The effect of fly ash on the expansion resulting from the alkali-aggregate reaction is also considered. To determine the variations in properties which may be expected and the effect of these variations on the behavior of the fly ash in mortar, 34 fly ashes from 19 different sources were examined.

The results of the tests indicate that generally the strength developed in portland cement-fly ash mortars is related to the carbon content of the fly ash, the fineness of fly ash as measured by the amount passing the No. 325 sieve, and the water required for mortars containing fly ash as compared to the water required for similar mortars without fly ash. No well-defined relation between the individual inorganic constituents of the fly ash and the development of strength of the mortar was obtained.

These tests also indicate that the effect on the strength of mortar of replacing a portion of the cement with fly ash varies according to the cement used.

Tests of mortar bars show that fly ash may be used to inhibit the expansion resulting from the alkali-aggregate reaction. These tests indicate that the amount of fly ash required to reduce expansion below a safe limit varies for different fly ashes.

Fly ash is a powdery residue resulting from the burning of pulverized coal. In modern power plants this material is collected at the entrance to the stack by electrical or mechanical precipitators to prevent pollution of the air. Fly ash is usually finer than portland cement and consists mostly of small spheres of glassy compounds of complex chemical composition together with miscellaneous materials such as quartz, feldspar, iron oxides, and carbon.[3] Photomicrographs of two fly ashes are shown in Fig. 1.

During recent years there has been a growing interest by both governmental and industrial organizations in the use

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

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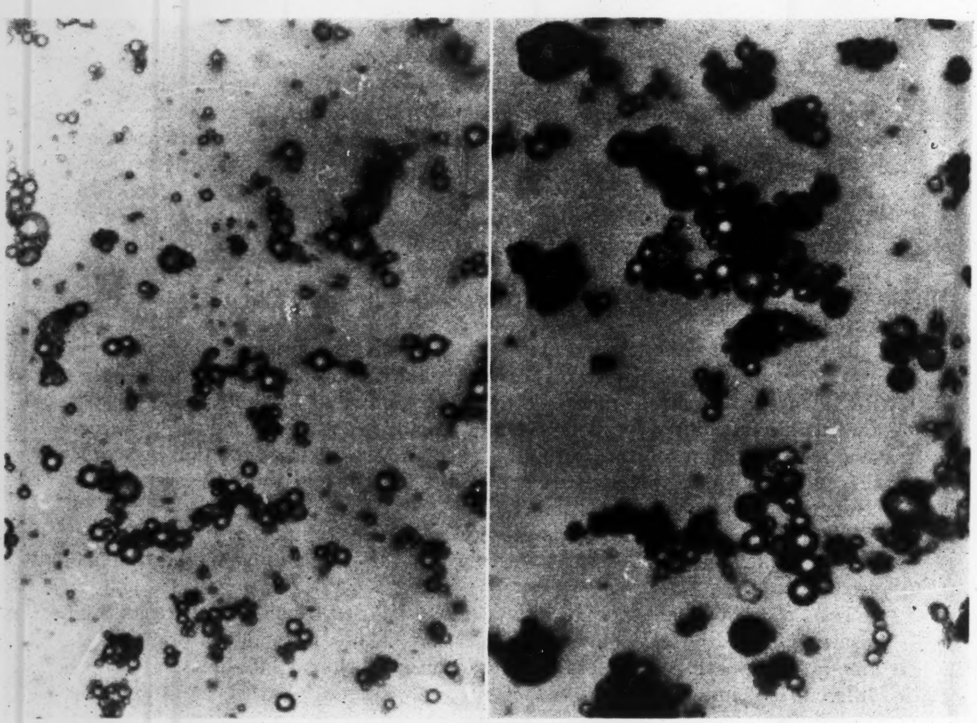
[2] Chemist, Physical Research Branch, Bureau of Public Roads.

[3] A discussion of the nature and source of these materials and the chemical changes that occur during the burning of the coal is included as Appendix II of this paper.

of fly ash as an additive to concrete. A number of reports have been published which indicate that beneficial effects may be obtained when fly ash is used to replace a portion of either the sand or the cement (**1, 2, 3, 4**).[4] In an investigation of concrete for the Hungry Horse Dam, Blanks (**1**) reported that fly ash improved the workability and decreased the bleeding of plastic concrete. The segregation of aggregates in concrete was also decreased. During the hardening of concrete it was found that a cement-fly ash combination produced less rise in temperature than an equal amount of portland cement. Other beneficial effects were stated to be decreased permeability, better resistance to sulfate attack, and reduced expansion resulting from the reaction between the alkalis of the cement and certain types of aggregate.

Fly ash has been used most extensively in mass concrete, particularly large dams, where many of the changes it produces in concrete are of value. As fly ash is less expensive than cement, its use to replace a portion of the cement has resulted in significant savings in the cost of certain projects. The possibility of reducing the cost of concrete for highway construction is responsible for much of the current interest by highway agencies in the use of fly ash. Interest has also been shown in its use at loca-



Fly Ash No. 1, 0.2 per cent carbon

Fly Ash No. 15, 5.5 per cent carbon

FIG. 1.—Photomicrographs of Fly Ash (×450).

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[4] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1202.

tions where the alkali-aggregate reaction is a problem.

Regardless of the primary reason for using fly ash in concrete, the effect of the admixture on the strength of the concrete must be considered. When fly ash is used to replace a portion of the cement, the strength of the resulting concrete is usually reduced at early ages but with adequate curing it may eventually equal or exceed that of concrete without fly ash (1, 2). Fly ash contributes to the strength of concrete at later ages because it is a pozzolanic material. A pozzolan is defined in ASTM Standard Definitions of Terms Relating to Hydraulic Cement[5] as "a siliceous or siliceous and aluminous material, which in itself possesses little or no cementitious value but will, in finely divided form and in the presence of moisture, chemically react with calcium hydroxide at ordinary temperatures to form compounds possessing cementitious properties." The reaction between fly ash and the lime released by the hydration of cement develops slowly and a minimum of 30 days may be required before the strength developed in concrete by this reaction will compensate for the initial loss in strength resulting from the lower cement content.

Fly ashes from different sources vary considerably in both chemical composition and physical properties due to differences between the coals from which they are derived and the conditions of burning. Consequently, it is to be expected that their behavior in concrete will not be uniform. This investigation was conducted primarily to study the pozzolanic behavior of fly ash as shown by its effect on the strength of cement mortar. The strengths developed in mortars containing fly ash were used to study the relations between pozzolanic activity and the various chemical and physical properties of fly ash. Comparisons were also made between the effect of fly ash on the strength of mortar and the results of other tests involving reaction of fly ash with lime or sodium hydroxide. At the time this investigation was undertaken, it was generally recognized that finely divided, low-carbon fly ashes were useful as admixtures to concrete. However, there was no general agreement as to the limits which should be placed on these properties or the extent to which these properties alone could be relied upon for selecting fly ashes. It was hoped that the information obtained in this investigation would provide a suitable basis for the selection of fly ashes for use in concrete. A study was also made of the effectiveness of fly ash in reducing expansion resulting from the alkali-aggregate reaction. These results were considered separately from the general behavior of fly ash as a pozzolanic material.

To have fly ashes with a wide range of physical and chemical properties represented in this investigation, 34 samples produced by 12 different concerns at 19 individual plants were obtained. Fly ashes from the sources represented by samples 1, 3, 5, and 16 had been used in concrete with good results, but the use or quality of the other samples were not known. Four of the samples (1, 3, 14, and 29) were also used in an investigation in which the effect of fly ash was determined directly on concrete specimens. The results of that study are reported in a companion article.[6]

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[5] Definition of terms as applied to Hydraulic Cement (C 219 – 55), 1955 Book of ASTM Standards, Part 3, p. 201.

[6] Fly ashes 1, 3, 14, and 29 are identified as A, B, X, and Y, respectively, in the report by Timms and Grieb, "Use of Fly Ash in Concrete," presented at the 59th Annual Meeting, Am. Soc. Testing Mats., Atlantic City, June 17–22 (1956).

CHEMICAL AND PHYSICAL PROPERTIES OF FLY ASH SAMPLES

The results of chemical analyses showing the principal constituents of the samples of fly ash used in this investigation are given in Table I. In recognition of the importance of the carbon content of the fly ash, identification numbers were assigned to the samples on the basis of increasing amounts of carbon.

The carbon in fly ash represents unburned portions of the coal and varies with the conditions of burning at the plant. Some fly ashes are produced which contain more carbon than any of the samples listed in Table I, but such materials are not likely to be considered

TABLE I.—CHEMICAL ANALYSIS OF FLY ASH (OVEN DRY BASIS).
All values are in per cent.

Identification	Loss on Ignition, 600 C	Carbon	SiO_2	Fe_2O_3	Al_2O_3	CaO	MgO	Na_2O	K_2O	Total Alkalis as Na_2O	SO_3	CO_2
No. 1	1.2	0.2	47.1	19.2	18.2	7.0	1.1	1.80	2.15	3.21	2.8	0.01
No. 2	1.0	0.5	48.4	19.7	17.9	5.2	1.2	2.10	2.20	3.55	2.3	0.03
No. 3	1.2	0.6	49.2	16.2	19.9	5.5	1.4	2.00	2.35	3.55	2.7	0.04
No. 4	1.7	0.7	39.1	31.3	17.6	5.2	0.9	0.35	1.92	1.61	1.5	0.08
No. 5	3.0	2.3	47.2	19.1	19.3	5.2	1.3	1.22	2.38	2.79	1.9	0.11
No. 6	4.1	2.4	46.7	16.4	20.3	6.4	1.1	0.50	2.00	1.82	2.1	0.64
No. 7	3.8	3.5	40.4	29.5	20.0	1.7	1.2	0.35	2.17	1.78	1.0	0.01
No. 8	4.1	3.5	47.1	16.3	22.2	4.5	1.1	1.00	2.50	2.64	1.2	0.07
No. 9	4.2	3.9	36.5	27.1	19.7	7.8	1.3	0.75	1.28	1.59	1.4	0.07
No. 10	4.0	3.9	38.5	25.4	17.9	8.3	1.0	0.40	2.00	1.72	2.3	0.13
No. 11	4.2	3.9	48.3	18.0	23.9	1.8	1.1	0.40	2.00	1.72	0.5	0.01
No. 12	5.0	4.5	51.9	12.3	23.7	2.3	1.1	0.40	2.20	1.85	0.4	0.02
No. 13	4.7	4.8	45.3	19.3	26.1	1.6	0.8	0.22	1.80	1.40	0.3	0.03
No. 14	5.4	5.0	41.2	20.6	22.1	6.0	1.2	1.00	1.42	1.93	0.9	0.04
No. 15	5.7	5.5	38.9	24.1	20.7	6.4	1.2	0.83	1.28	1.67	1.0	0.10
No. 16	7.5	6.1	44.8	11.2	18.4	11.6	1.1	0.92	2.22	2.38	2.0	0.10
No. 17	6.9	6.4	42.2	19.4	22.4	4.3	1.2	0.67	1.82	1.87	1.0	0.05
No. 18	7.1	6.9	49.2	8.7	26.7	3.6	0.9	0.58	2.38	2.15	0.6	0.03
No. 19	7.4	6.9	45.3	16.9	26.0	1.4	0.8	0.25	2.00	1.57	0.3	0.01
No. 20	8.6	7.8	48.2	17.7	16.1	5.2	1.2	0.30	2.25	1.78	0.3	0.10
No. 21	8.6	7.9	51.2	8.5	25.6	1.6	0.9	0.48	2.75	2.29	0.3	0.06
No. 22	9.2	8.2	44.2	19.9	14.6	7.6	1.3	0.32	1.97	1.62	1.0	0.14
No. 23	11.2	8.4	40.4	16.3	18.3	8.5	1.0	0.70	1.62	1.77	1.7	1.26
No. 24	9.2	8.9	45.3	18.6	22.4	1.5	0.9	0.67	1.42	1.60	0.3	0.03
No. 25	12.0	8.9	44.6	12.6	20.0	6.2	1.2	0.32	2.02	1.64	0.9	1.27
No. 26	9.2	9.1	48.9	8.8	28.3	1.2	0.8	0.52	2.20	1.97	0.4	0.02
No. 27	12.5	10.5	41.6	14.2	19.0	6.8	1.2	0.58	1.80	1.76	2.2	0.51
No. 28	11.2	10.6	46.7	12.9	24.7	1.1	0.9	0.50	2.05	1.85	0.4	0.02
No. 29	11.6	11.2	38.5	18.8	23.5	3.2	1.0	0.60	1.88	1.84	0.6	0.03
No. 30	16.9	11.9	45.8	6.6	13.5	12.0	2.7	1.61	0.50	1.94	0.8	1.61
No. 31	14.8	13.6	40.2	15.0	18.6	3.9	1.0	1.15	2.98	3.11	2.2	0.04
No. 32	14.5	14.3	44.1	10.9	25.5	1.7	0.7	0.47	2.00	1.79	0.2	0.04
No. 33	16.4	14.8	32.7	28.0	17.1	1.8	1.1	0.25	1.73	1.39	1.3	0.03
No. 34	18.0	15.6	40.1	15.2	17.5	3.5	1.1	0.70	1.80	1.88	1.8	0.15
Minimum	1.0	0.2	32.7	8.5	14.6	1.1	0.7	0.22	1.28	1.39	0.3	0.01
Maximum	18.0	15.6	51.9	31.3	28.3	12.0	1.4	2.10	2.98	3.55	2.8	1.61

for use in concrete. Although the loss on ignition of fly ash is generally considered to be closely related to the carbon content, Table I and Fig. 2 show that the loss can be considerably greater in some cases. Of these samples, five showed differences equal to or greater than 2.0 per cent, the greatest difference being 5.0 per cent for sample 30. These large differences usually occur when there is an appreciable amount of calcium hydroxide or carbonate present. At the ignition temperature used (600 C) these materials decompose to the oxide and either water or carbon dioxide. As the latter two constituents are volatile they increase the loss on ignition. Combined moisture from other minerals may also increase the loss.

It should be noted that on account of the differences between the values for loss on ignition and carbon content, specification limits for the maximum ignition loss will insure for all practical purposes, that the carbon content will be less than this and sometimes by a significant amount. The one exception to this general statement is the case of sample 13 where the carbon content exceeds the loss on ignition by 0.1 of a percentage point. This small difference is within the limits of experimental error.

It is possible that some of the samples tested contain small amounts of manganese, titanium, and possibly phosphorus. The titanium and phosphorus, if present, are included with the reported percentage of alumina (Al_2O_3). When manganese is present, most of it is included with the magnesia (MgO), and the balance with the lime (CaO). This manner of determination and reporting follows the usual practice in silicate analysis. It is believed that these minor constituents have no significance in this investigation. Details of the methods followed in making the chemical analyses are given in Procedure 1 of Appendix I to this paper.

The specific gravity and fineness for all samples of fly ash are given in Table II. Sample 30 had only 33.1 per cent passing the No. 200 sieve, and mortar tests were not made with this fly ash. The specific gravities of the fly ashes vary from 2.12 to 2.69 and are found to bear a general relation to the amounts of iron oxide present in the sample, high specific gravities being indicative of high iron contents.

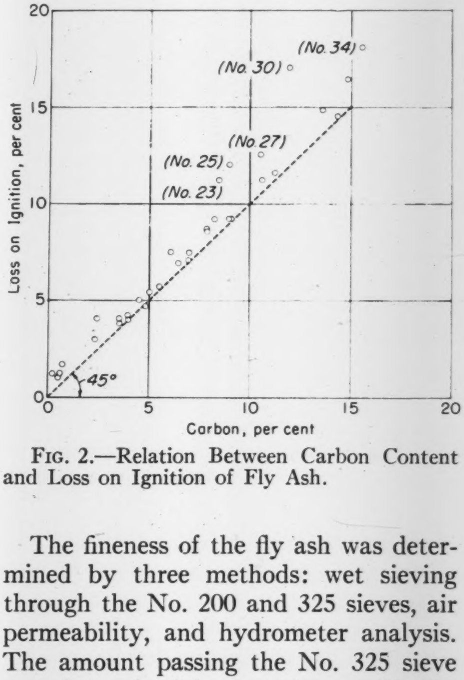

FIG. 2.—Relation Between Carbon Content and Loss on Ignition of Fly Ash.

The fineness of the fly ash was determined by three methods: wet sieving through the No. 200 and 325 sieves, air permeability, and hydrometer analysis. The amount passing the No. 325 sieve was determined in accordance with the procedure outlined in section 12 of the ASTM Method of Test for Fineness of Portland Cement by the Turbidimeter.[7] Specific surface by air permeability was determined by the ASTM Method of Test for Fineness of Portland Cement

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[7] Method of Test for Fineness of Portland Cement by the Turbidemeter (C 115 – 53), 1955 Book of ASTM Standards, Part 3, p. 152.

TABLE II.—SPECIFIC GRAVITY AND FINENESS OF FLY ASH SAMPLES.

Fly Ash Identification	Apparent Specific Gravity	Fineness				
		Amount Passing, per cent		Air Permeability Specific Surface, sq cm per g	Hydrometer Method	
		No. 200 Sieve	No. 325 Sieve		Specific Surface, sq cm per g	Amount Smaller than 0.03 mm, per cent
No. 1	2.49	96.8	93.2	3075	4050	76
No. 2	2.49	95.0	92.1	3295	5105	79
No. 3	2.52	98.6	95.2	4305	5570	86
No. 4	2.69	94.6	90.0	2550	3130	67
No. 5	2.46	97.4	95.3	3585	3115	87
No. 6	2.38	96.2	92.1	3960	3085	74
No. 7	2.62	94.4	91.7	3775	3290	76
No. 8	2.44	96.8	93.9	4290	3520	81
No. 9	2.65	95.6	92.9	3110	2475	75
No. 10	2.58	95.4	91.7	4250	4070	81
No. 11	2.36	88.2	83.2	3030	1390	47
No. 12	2.37	93.2	88.9	3580	3715	71
No. 13	2.32	82.0	75.8	2960	2025	48
No. 14	2.51	91.8	84.5	2565	2770	59
No. 15	2.50	85.8	81.3	2845	930	50
No. 16	2.39	95.2	93.2	5355	2080	94
No. 17	2.52	94.2	91.5	4020	2330	56
No. 18	2.31	92.4	89.3	4060	2565	68
No. 19	2.33	90.2	86.4	3830	2450	61
No. 20	2.40	90.8	81.6	2560	...	...
No. 21	2.12	57.2	56.2	2430	2155	31
No. 22	2.39	90.4	82.0	2985	2088	67
No. 23	2.30	85.4	76.6	3315	1260	67
No. 24	2.41	92.2	86.6	3140	1970	60
No. 25	2.30	77.0	74.8	4115	2225	47
No. 26	2.22	87.6	82.8	4405	3255	68
No. 27	2.31	82.8	82.5	4650	...	...
No. 28	2.24	85.0	81.9	4295	1965	50
No. 29	2.43	93.8	89.4	3220	2000	66
No. 30	2.20	33.1	...	...	...	...
No. 31	2.42	96.0	93.2	4795	2435	79
No. 32	2.25	87.2	80.9	4400	2250	51
No. 33	2.44	81.5	76.1	3860	1795	38
No. 34	2.29	81.0	78.1	4610	2230	62

by Air Permeability Apparatus.[8] The weight of sample used in this test was determined by trial as the weight necessary to obtain a hard, firm bed in the permeability cell. For many samples it was found necessary to use a porosity for the test bed of fly ash which differed considerably from the porosity used in the calibration of the apparatus. Fineness by the hydrometer method was determined in accordance with the Standard Methods of Mechanical Analysis of Soils[9, 10] using 125 ml of a 0.4*N* solution of sodium hexametaphosphate as the deflocculating agent. Dispersion cup A shown in Fig. 3 of the method was used. The initial weight of each sample was corrected for the amount of water-soluble material present in calculating the amount of sample smaller than each size.

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[8] Method of Test for Fineness of Portland Cement by Air Permeability Apparatus (C 204 – 55), 1955 Book of ASTM Standards, Part 3, p. 145.

The grain diameter accumulation curve obtained by the hydrometer analysis was used to calculate the specific surface based on the amounts of the sample estimated to have average diameters of 0.7, 2, 3, 4, 5, 6, 7, 8, 9, 12, 20, 30, 40, 50, and 60 μ. The grain diameter accumulation curves for three fly ashes are shown in Fig. 3. The curves for the remaining samples used in this investigation lie essentially within the area bounded by the curves for fly ashes Nos. 3 and 15, which have the highest and lowest specific surfaces respectively as calculated by this method. The curve for sample 28 is included to illustrate the discrepancy that is often shown between values for specific surface obtained by the air permeability and hydrometer methods. The specific surfaces by the air permeability method for fly ashes Nos. 3 and 28 were about 4300 sq cm per g, which classifies both as relatively fine materials. The grain diameter accumulation curves, however, are widely different. The specific surfaces for these two materials are also quite different when determined by the hydrometer method, being 5570 sq cm per g for sample No. 3 and 1965 sq cm per g for sample No. 28.

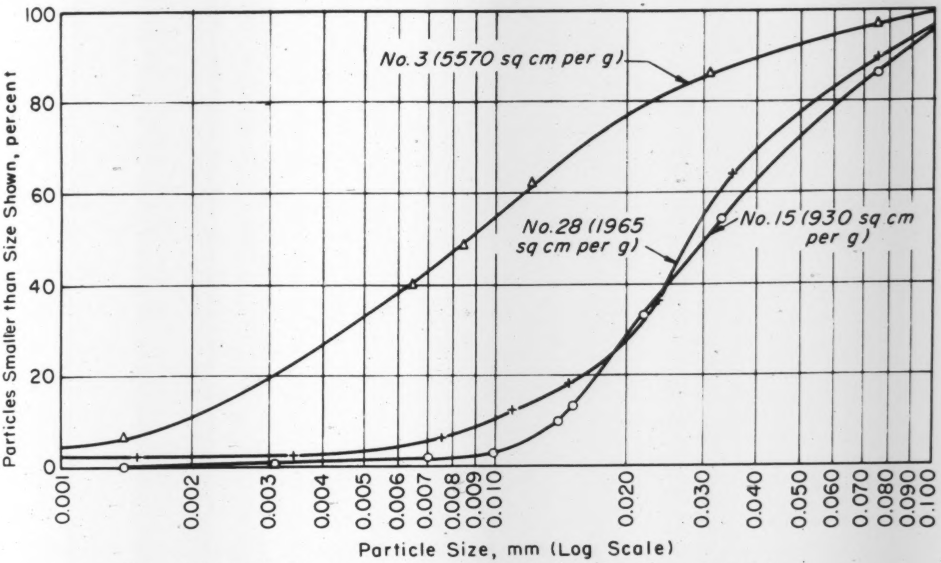

FIG. 3.—Grain Diameter Accumulation Curves for Three Fly Ashes.

In Table III, average values for fineness as determined by each method are shown for the samples of fly ash which have been grouped with respect to carbon content. Samples Nos. 20, 27, and 30 are not included, as these materials were not tested by all three methods for fineness. For the samples considered

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[9] Standard Methods of Mechanical Analysis of Soils (T 88 – 54) 1955 Book of Standard Specifications for Highway Materials and Methods of Sampling and Testing (AASHO), Part II, p. 257.

[10] Tentative Method of Grain-Size Analysis of Soils (D 422 – 54 T), 1955 Book of ASTM Standards, Part 3, p. 1756.

TABLE III.—AVERAGE FINENESS OF FLY ASHES BY GROUPS.

Range of Carbon Content, per cent	Number of Samples Represented	Average Fineness Values			
		Amount Passing No. 325 Sieve, per cent	Air Permeability, Specific Surface, sq cm per g	Hydrometer Method	
				Specific Surface, sq cm per g	Amount Smaller than 0.03 mm, per cent
0–4	11	91.9	3570	3530	75
4–8	9	83.0	3520	2340	60
8–12	7	82.0	3640	2110	61
12–15.6	4	81.0	4420	2180	58

here, those with the most carbon were indicated to be the finest by the air permeability method. Determinations of fineness by both the No. 325 sieve and the hydrometer, however, showed that the finest fly ashes were those containing relatively little carbon.

These data indicate that the determination of the fineness of fly ash by the air permeability method is likely to furnish misleading information. A possible cause of this can be seen by examining the photomicrographs of fly ash in Fig. 1. The transparent, spherical particles are composed chiefly of the inorganic portion of each fly ash. The dark, irregularly-shaped particles present in fly ash No. 15 are composed of carbon and appear to be highly porous. It is believed that the results of the air permeability method are influenced by both the external and internal surfaces of particles of carbon. Consequently, increase in the carbon content of fly ash may be accompanied by an increase in total surface area even though the external surface area and the actual fineness of the material decrease.

TABLE IV.—PROPERTIES OF CEMENTS USED IN PREPARING MORTARS CONTAINING FLY ASH.

	Cement A	Cement B	Cement C
Chemical Analysis:			
Silicon dioxide (SiO_2), per cent	21.8	22.3	21.7
Aluminum oxide (Al_2O_3), per cent	5.7	5.4	6.1
Ferric oxide (Fe_2O_3), per cent	2.5	2.4	2.8
Calcium oxide (CaO), per cent	61.9	66.1	64.9
Magnesium oxide (MgO), per cent	2.6	1.0	1.2
Sulfur trioxide (SO_3), per cent	1.9	1.7	1.6
Loss on ignition, per cent	2.1	1.2	1.2
Insoluble residue, per cent	0.19	0.12	...
Sodium oxide (Na_2O), per cent	0.33	0.04	0.13
Potassium oxide (K_2O), per cent	0.86	0.15	0.72
Equivalent alkali as Na_2O, per cent	0.90	0.13	0.60
Compound Composition:			
Tricalcium silicate (C_3S), per cent	39	55	50
Dicalcium silicate (C_2S), per cent	33	22	25
Tricalcium aluminate (C_3A), per cent	11	10	12
Physical Properties:			
Fineness (Wagner turbidimeter), sq cm per g	1785	1625	1875
Normal consistency, per cent	24.5	25.0	25.5
Air content of mortar, per cent	10.0	6.8	7.8
Compressive strength,[a] psi			
7 days	2260	2960	4325
28 days	3560	4725	6000
Time of set: Initial, hr—min	2–35	4–10	4–0
Final, hr—min	4–0	6–25	5–5

[a] ASTM Method C 109, see footnote [11].

TABLE V.—WATER REQUIREMENT RATIO OF CEMENT - FLY ASH MORTARS, PER CENT.

Fly Ash Identification	Water Requirement Ratio, Based on Control Mortar											
	10 per cent Replacement			20 per cent Replacement			35 per cent Replacement			50 per cent Replacement		
	Cement			Cement			Cement			Cement		
	A	B	C	A	B	C	A	B	C	A	B	C
1:2.75 MORTAR												
No. 1	96	04	94	93	93	92	90	90	89	86	86	85
No. 2	100	100	101	97	96	99	92	91	94	88	87	89
No. 3	96	94	94	93	93	92	90	90	89	87	87	85
No. 4	103	101	101	97	99	99	92	97	97	91	96	95
No. 5	101	100	101	97	97	97	94	94	93	90	91	90
No. 6	101	101	101	99	99	99	97	100	97	101	101	101
No. 7	104	103	103	104	103	102	103	101	100	103	103	100
No. 8	101	101	102	100	101	100	97	99	97	94	94	95
No. 9	100	101	102	100	101	102	99	100	100	97	99	98
No. 10	100	99	102	100	100	102	100	101	103	100	101	103
No. 11	104	103	103	101	104	101	104	101	101	103	101	103
No. 12	101	100	102	98	100	100	97	99	98	94	97	97
No. 13	100	100	102	101	103	105	103	104	106	104	109	111
No. 14	101	100	100	101	100	100	101	100	100	103	100	101
No. 15	101	103	103	104	104	105	106	107	106	107	109	109
No. 16	103	100	101	100	97	99	97	94	96	94	93	94
No. 17	98	100	101	97	101	100	97	101	101	97	101	101
No. 18	102	99	103	102	100	101	102	100	101	102	100	101
No. 19	98	103	103	98	103	105	98	103	105	102	106	108
No. 20	100	100	100	98	101	100	98	100	101	98	101	101
No. 21	102	100	101	106	106	108	112	113	116	118	122	125
No. 22	102	103	103	103	106	106	108	110	111	111	113	124
No. 23	100	101	100	100	103	101	102	106	104	105	109	107
No. 24	100	100	98	102	103	101	103	104	103	106	106	106
No. 25	101	101	101	103	104	104	109	109	109	116	115	116
No. 26	100	103	103	102	107	108	105	109	112	109	113	116
No. 27	107	107	107	109	109	109	118	118	115	125	125	119
No. 28	104	104	101	106	104	106	110	109	109	118	115	115
No. 29	101	101	101	103	103	103	106	103	104	108	106	106
No. 31	100	99	99	99	99	97	97	99	97	99	100	99
No. 32	103	103	101	104	106	104	109	109	109	113	113	113
No 33	101	104	101	106	109	107	113	119	117	122	126	125
No. 34	109	109	109	118	116	116	125	125	125	134	134	137
1:2 MORTAR												
No. 1	99	97	97	94	93	93	89	89	89	84	84	85
No. 3	96	99	99	92	93	94	86	90	92	82	86	87
No. 14	100	101	100	102	100	100	101	100	100	101	100	100
No. 29	100	101	100	100	103	101	105	106	104	110	109	107
1:3.5 MORTAR												
No. 1	99	97	97	95	95	93	91	92	91	88	89	88
No. 3	97	97	96	95	95	93	91	92	91	88	89	87
No. 14	101	97	100	101	99	100	101	99	100	101	100	100
No. 29	101	101	101	104	103	103	107	105	104	108	107	105

TABLE VI.—COMPRESSIVE STRENGTH OF CEMENT-FLY ASH MORTARS PREPARED WITH CEMENT A—MOIST AIR STORAGE AT 73 F.

Fly Ash Identification	Compressive Strength of Control Mortar, psi (without Fly Ash)[a]			Compressive Strength Ratio of Mortars Containing Fly Ash,[b] per cent											
				10 per cent Replacement			20 per cent Replacement			35 per cent Replacement			50 per cent Replacement		
	28 days	91 days	365 days	28 days	91 days	365 days	28 days	91 days	365 days	28 days	91 days	365 days	28 days	91 days	365 days
1:2.75 MORTAR															
No. 1	3180	3840	4240	92	94	113	90	100	121	82	101	110	72	84	90
No. 2	3760	4440	4940	100	98	112	95	104	117	84	100	106	80	94	98
No. 3	3180	3840	4240	97	99	111	98	110	126	97	116	123	95	105	111
No. 4	3420	3770	4440	101	108	118	96	106	119	82	103	112	64	93	90
No. 5	3000	3630	4120	112	117	121	102	117	124	102	127	135	93	112	115
No. 6	3360	3780	4290	96	100	101	91	99	113	82	94	103	67	76	79
No. 7	3420	3770	4440	108	115	118	99	116	126	86	112	113	65	89	95
No. 8	3420	3770	4440	107	115	125	101	120	130	96	126	131	87	114	122
No. 9	3760	4440	4940	97	100	102	86	97	108	76	98	111	62	89	102
No. 10	3540	4120	4710	96	99	97	95	99	111	83	99	108	72	82	87
No. 11	3440	4110	4400	96	101	110	82	90	105	65	87	103	51	74	87
No. 12	3680	4170	4590	94	103	112	88	106	113	83	105	112	67	93	97
No. 13	3540	4120	4710	95	96	99	75	84	90	64	84	88	45	61	71
No. 14	3330	3940	4550	90	93	99	75	86	94	60	77	89	47	68	77
No. 15	3760	4440	4940	89	93	93	81	89	95	67	80	89	51	68	77
No. 16	3440	4110	4400	103	102	112	95	114	127	95	121	138	91	114	124
No. 17	3520	3830	4580	104	113	112	96	107	108	87	117	118	75	97	102
No. 18	3740	4450	4750	95	100	103	90	103	115	86	108	121	64	82	105
No. 19	3520	3830	4580	95	105	98	88	108	108	72	96	100	58	86	96
No. 20	3740	4450	4750	94	101	101	85	96	108	66	81	103	45	63	78
No. 21	3740	4450	4750	78	82	84	72	75	81	53	60	66	30	44	48
No. 22	3520	3830	4580	96	99	99	86	100	105	68	88	95	46	67	73
No. 23	3800	4020	4490	88	96	98	75	88	96	54	75	85	42	56	65
No. 24	3520	3830	4580	92	100	100	88	101	98	71	96	94	56	81	88
No. 25	3360	3780	4290	92	93	93	84	90	89	62	74	76	42	52	52
No. 26	3520	3830	4580	94	108	107	92	109	110	69	93	94	54	81	84
No. 27	3000	3630	4120	98	94	97	83	85	89	62	76	82	41	51	52
No. 28	3440	4110	4400	93	101	111	86	98	105	66	85	95	44	65	81
No. 29	3330	3940	4550	84	91	95	73	81	93	60	76	84	46	60	74
No. 31	3520	3990	4560	108	111	114	91	109	116	80	109	115	70	99	105
No. 32	3520	3990	4560	95	101	98	78	89	93	64	83	92	39	59	74
No. 33	3520	3990	4560	94	90	93	81	91	89	60	76	79	41	56	64
No. 34	3000	3630	4120	93	100	97	85	93	92	67	82	80	44	56	58
Average				96	101	104	87	99	106	74	94	102	59	78	85
1:2 MORTAR															
No. 1	4670	5340	5890	102	102	113	99	108	124	97	115	119	92	104	114
No. 3	4560	5490	6510	102	103	108	103	115	115	109	121	114	114	117	109
No. 14	4960	5800	6840	96	102	104	80	87	94	63	80	86	54	73	78
No. 29	4960	5800	6840	95	104	102	75	92	100	64	81	91	44	64	75
Average				99	103	107	89	100	108	83	99	102	76	90	94
1:3.5 MORTAR															
No. 1	2540	2950	3170	97	108	117	83	97	124	73	98	121	61	88	103
No. 3	2540	2950	3170	92	100	113	92	107	133	91	110	136	82	103	117
No. 14	2540	2950	3170	88	97	103	78	67	76	57	82	108	42	68	98
No. 29	2330	2920	3000	105	106	133	95	97	122	72	85	109	51	61	88
Average				96	103	116	87	92	114	73	94	118	59	80	102

[a] Each value is an average of three 2-in. cubes.

[b] Reported as a percentage of the strength of control mortar.

TABLE VII.—COMPRESSIVE STRENGTH OF CEMENT-FLY ASH MORTARS PREPARED WITH CEMENT B—MOIST AIR STORAGE AT 73 F.

Fly Ash Identification	Compressive Strength of Control Mortar, psi (without Fly Ash)[a]			Compressive Strength Ratio of Mortars Containing Fly Ash,[b] per cent											
				10 per cent Replacement			20 per cent Replacement			35 per cent Replacement			50 per cent Replacement		
	28 days	91 days	365 days	28 days	91 days	365 days	28 days	91 days	365 days	28 days	91 days	365 days	28 days	91 days	365 days
1:2.75 Mortar															
No. 1	5130	6260	7030	92	92	98	82	93	102	66	84	97	51	75	87
No. 2	5020	6360	6560	108	105	113	92	97	107	67	85	97	63	79	87
No. 3	5130	6260	7030	95	103	105	93	101	113	75	93	107	69	89	98
No. 4	4840	6000	6440	99	83	103	88	95	103	72	89	105	46	63	76
No. 5	5150	6190	6780	94	100	108	88	97	113	69	91	111	55	76	98
No. 6	5560	6730	6930	83	83	94	70	76	88	54	76	83	35	50	66
No. 7	5290	6240	6390	90	92	99	78	88	101	65	84	94	45	67	92
No. 8	5290	6240	6390	88	95	104	81	94	107	74	97	121	45	70	102
No. 9	4840	6000	6440	99	101	105	81	89	104	56	70	92	46	68	87
No. 10	4840	6000	6440	105	107	109	87	93	105	66	82	97	46	67	77
No. 11	5220	6410	7040	83	91	91	67	80	90	48	65	77	33	50	64
No. 12	5020	6360	6560	97	99	101	80	82	94	63	78	98	53	72	93
No. 13	4840	6000	6440	99	100	101	77	86	94	51	66	76	35	50	60
No. 14	4930	6290	6620	92	86	95	75	79	93	55	69	85	36	51	62
No. 15	4840	6000	6440	91	98	102	72	81	93	48	63	80	28	40	62
No. 16	5220	6410	7040	87	95	97	74	86	96	57	78	96	47	72	92
No. 17	4760	5540	5870	92	100	108	85	101	114	65	86	101	43	64	86
No. 18	5030	6040	6540	87	95	100	79	90	104	58	78	95	40	61	82
No. 19	4910	5770	6260	87	100	104	79	89	95	58	74	86	37	51	71
No. 20	4760	5540	5870	86	97	107	75	85	100	54	70	88	38	52	73
No. 21	5030	6040	6540	73	81	80	58	67	69	33	45	51	20	33	41
No. 22	4760	5540	5870	86	94	103	65	78	91	51	68	85	31	45	66
No. 23	4840	5900	6590	89	92	93	68	73	81	47	60	72	29	42	51
No. 24	4840	5900	6590	97	93	98	72	90	92	59	73	82	37	51	67
No. 25	5560	6730	6930	75	82	72	71	75	80	49	57	65	26	39	49
No. 26	4840	5900	6590	94	98	98	76	88	90	54	74	83	37	56	64
No. 27	5150	6190	6780	85	78	90	66	71	84	43	56	65	28	40	47
No. 28	5220	6410	7040	87	92	97	70	81	85	44	61	69	26	41	55
No. 29	4930	6290	6620	87	89	100	73	77	91	53	60	75	33	44	57
No. 31	5220	6640	6930	92	95	106	77	91	105	60	71	96	37	51	73
No. 32	5220	6640	6930	84	85	93	63	72	78	44	57	70	25	36	48
No. 33	5220	6640	6930	83	88	93	64	73	79	40	53	61	23	34	44
No. 34	5150	6190	6780	92	92	96	68	70	87	41	55	62	28	41	49
Average				90	94	99	76	84	95	56	72	86	39	55	70
1:2 Mortar															
No. 1	8440	10390	10970	87	93	99	82	89	96	70	85	99	60	71	81
No. 3	8560	10260	10960	94	97	108	87	100	108	82	95	107	76	83	91
No. 14	8440	10390	10970	90	91	101	71	82	92	56	64	80	40	49	62
No. 29	8440	10390	10970	92	93	101	76	80	89	53	60	74	33	43	57
Average				91	94	102	79	88	96	65	76	90	52	62	73
1:3.5 Mortar															
No. 1	3260	4270	4500	85	98	113	71	90	103	58	79	100	42	65	94
No. 3	3260	4270	4500	86	93	107	77	88	106	63	78	107	52	71	97
No. 14	3260	4270	4500	75	84	93	60	70	87	43	60	74	28	44	64
No. 29	3320	4140	4540	86	90	96	71	79	90	51	64	78	31	43	53
Average				83	91	102	70	82	96	54	70	90	38	56	77

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[a] Each value is an average of three 2-in. cubes.

[b] Reported as a percentage of strength of control mortar.

TABLE VIII.—COMPRESSIVE STRENGTH OF CEMENT - FLY ASH MORTARS PREPARED WITH CEMENT C—MOIST AIR STORAGE AT 73 F.

Fly Ash Identification	Compressive Strength of Control Mortar, psi (without Fly Ash)[a]			Compressive Strength Ratio of Mortars Containing Fly Ash,[b] per cent											
				10 per cent Replacement			20 per cent Replacement			35 per cent Replacement			50 per cent Replacement		
	28 days	91 days	365 days	28 days	91 days	365 days	28 days	91 days	365 days	28 days	91 days	365 days	28 days	91 days	365 days
1:2.75 Mortar															
No. 1	5920	6240	6590	90	96	103	79	89	102	64	87	107	58	79	98
No. 2	5390	5670	6070	96	97	101	89	99	108	76	94	106	69	90	104
No. 3	5920	6240	6590	92	99	102	89	102	114	76	98	113	70	91	101
No. 4	5490	5870	5860	90	96	103	75	87	99	67	82	96	50	68	89
No. 5	5670	5740	5770	94	99	114	89	104	119	74	100	117	66	90	113
No. 6	5250	5270	5530	92	100	104	81	95	100	61	83	87	48	64	71
No. 7	5490	5870	5860	94	100	111	81	94	105	72	87	102	52	70	88
No. 8	5490	5870	5860	101	106	117	89	107	118	83	105	117	62	81	108
No. 9	4900	5420	5790	107	104	103	97	106	106	77	94	107	53	68	83
No. 10	5490	5870	5860	96	104	102	85	95	99	69	85	87	52	74	87
No. 11	6100	5970	6190	89	104	109	77	92	102	58	80	89	36	58	79
No. 12	5390	5670	6070	98	102	108	93	106	112	73	86	105	59	79	98
No. 13	5490	5870	5860	93	96	97	76	79	95	...	...	...	...	...	...
No. 13	4900	5420	5790	...	...	...	...	...	...	65	73	82	43	55	68
No. 14	5440	5610	5640	90	92	94	79	87	98	59	75	86	43	59	72
No. 15	4900	5420	5790	98	95	94	84	92	93	65	75	83	41	54	69
No. 16	6100	5970	6190	88	98	102	80	98	108	71	90	106	58	83	108
No. 17	5080	5360	5650	104	107	111	92	102	106	80	93	105	57	78	94
No. 18	5410	5220	5750	103	107	115	97	112	112	73	98	108	55	76	88
No. 19	5080	5360	5650	99	103	103	85	98	102	57	72	81	41	67	77
No. 20	5410	5220	5750	93	104	101	82	95	104	60	77	89	42	61	72
No. 21	5410	5220	5750	85	95	88	67	79	75	42	58	57	25	38	42
No. 22	5080	5360	5650	93	96	99	78	93	97	56	69	80	36	47	63
No. 23	5570	5640	5940	87	92	93	67	79	86	49	67	78	31	48	62
No. 24	5570	5640	5940	85	90	87	72	82	90	59	74	91	36	57	75
No. 25	5250	5270	5530	91	98	95	76	84	83	58	71	75	37	50	55
No. 26	5080	5360	5650	92	97	97	77	86	91	57	73	81	37	54	65
No. 27	5670	5740	5770	90	96	97	72	77	82	49	59	65	38	50	55
No. 28	6100	5970	6190	86	95	95	72	85	88	54	69	77	36	54	67
No. 29	5440	5610	5640	89	91	109	80	87	101	58	68	85	38	50	67
No. 31	5540	5500	5490	98	105	111	79	93	106	65	85	109	48	74	97
No. 32	5540	5500	5490	91	97	102	69	78	84	47	63	79	28	42	58
No. 33	5540	5500	5490	94	97	102	68	75	75	44	56	64	31	42	50
No. 34	5670	5740	5770	91	100	99	74	84	86	49	61	67	34	46	51
Average				93	99	102	80	92	98	63	79	90	46	64	78
1:2 Mortar															
No. 1	8680	8940	9740	84	91	95	80	91	99	70	87	101	60	79	86
No. 3	7490	8120	9420	100	104	104	99	114	109	92	113	110	85	102	98
No. 14	8680	8940	9740	94	101	101	81	93	97	60	76	84	44	61	71
No. 29	8680	8940	9740	92	98	99	78	88	92	58	71	79	34	48	61
Average				92	98	100	84	96	99	70	87	94	56	72	79
1:3.5 Mortar															
No. 1	4140	4050	4270	87	103	105	76	98	103	66	90	110	57	80	103
No. 3	4140	4050	4270	92	101	105	87	108	116	72	103	125	66	99	119
No. 14	4140	4050	4270	90	99	96	78	91	99	53	72	87	37	57	80
No. 29	3890	4090	4160	93	96	98	75	86	90	55	68	80	36	49	65
Average				90	100	101	79	96	102	62	83	100	49	71	92

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[a] Each value is an average of three 2-in. cubes.

[b] Reported as a percentage of the strength of control mortar.

COMPRESSIVE STRENGTH TESTS OF MORTAR CONTAINING FLY ASH

The test specimens for determining the effect of fly ash on the compressive strength of mortar were 2-in. cubes and were made both from a control mortar containing no fly ash and test mortars in which various amounts of the same cement were replaced with fly ash on a solid volume basis. Each fly ash was used to replace 10, 20, 35, and 50 per cent of the cement in a 1:2.75 mix by weight using graded Ottawa sand. Control specimens were prepared each day that specimens containing fly ash were made. All cement-fly ash combinations were repeated with each of three cements whose properties are given in Table IV.

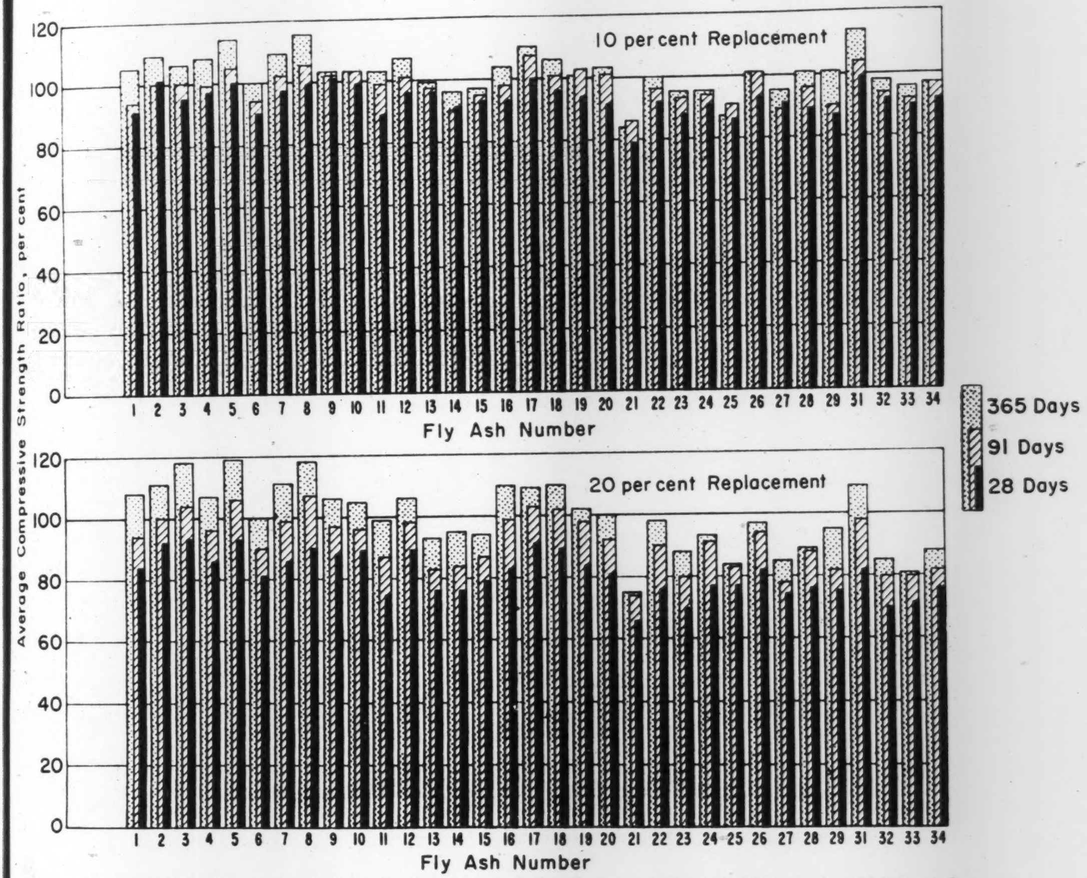

FIG. 4.—Development of Compressive Strength in Cement - Fly Ash Mortar for 33 Fly Ashes.

Note: Values plotted are averages of the compressive strength ratios obtained with cements A, B and C in a 1:2.75 mortar.

Samples arranged from left to right in order of increasing carbon content.

The mixing of the mortars and molding of the specimens were performed in accordance with the ASTM Method of Test for Compressive Strength of Hydraulic Cement Mortars.[11] As specified in this procedure a uniform consistency of all mortars was maintained by adjusting the amount of water added to each mix. The effect of a particular fly ash on the amount of mixing water required can be indicated conveniently by calculating the ratio of the amount of water required by the cement - fly ash mortar to that required by the control mortar. This ratio will be referred to subsequently as "water requirement ratio." After removal from the molds at an age of 24 hr, all specimens were stored at 73 F in moist air in tightly covered containers until tested at ages of 28, 91, and 365 days.

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[11] Method of Test for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. Cube Specimens) (C 109 – 49), 1949 Book of ASTM Standards, Part 3, p. 92.

The water requirement ratios used for preparing the mortars containing fly ash are given in Table V. The results of the tests for compressive strength of these mortars are given in Tables VI, VII, and VIII. For the convenience of the reader, the actual average strengths obtained in tests of the control mortars (without fly ash) are given, but the strengths of the mortars containing fly

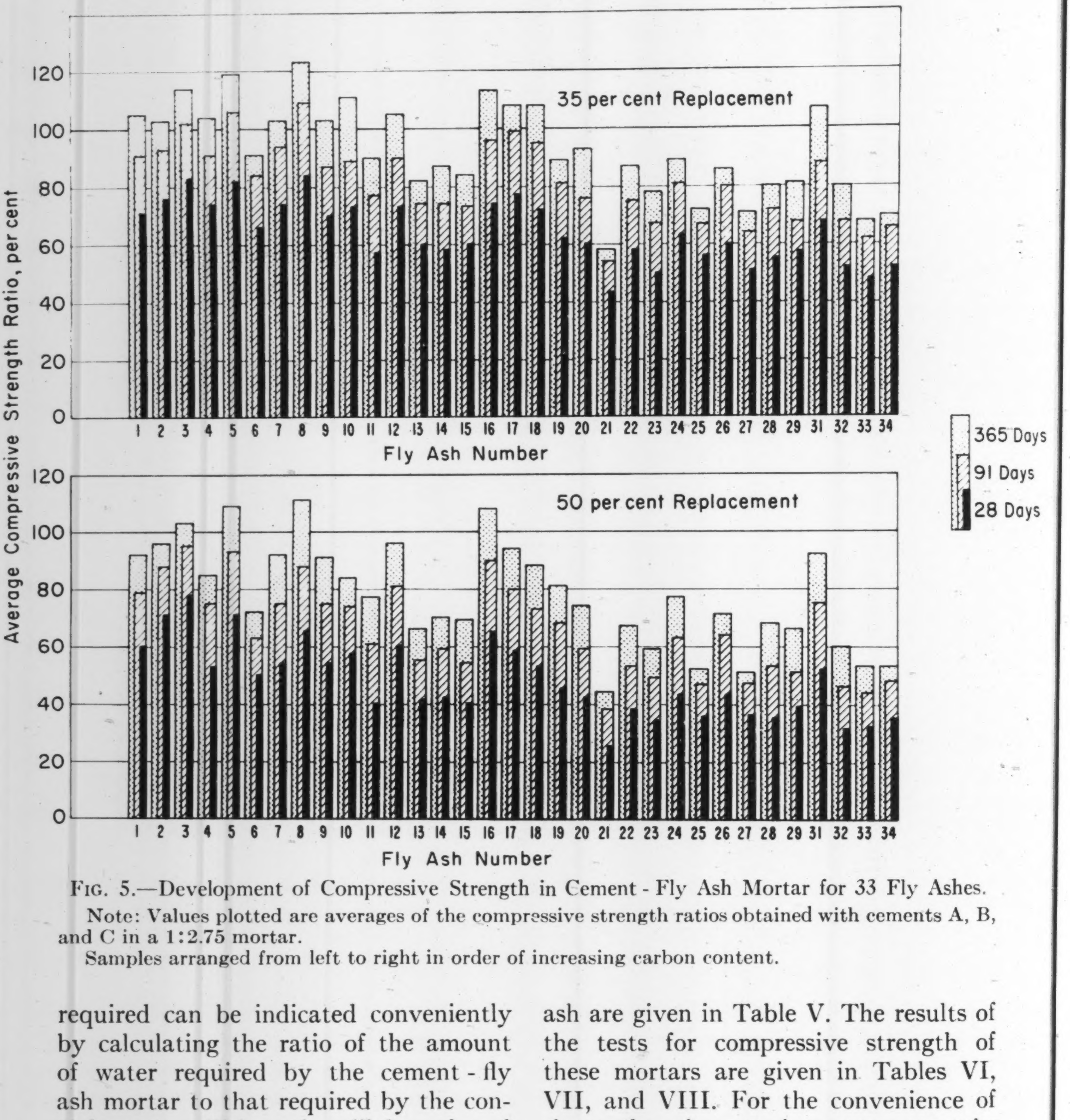


FIG. 5.—Development of Compressive Strength in Cement - Fly Ash Mortar for 33 Fly Ashes.

Note: Values plotted are averages of the compressive strength ratios obtained with cements A, B, and C in a 1:2.75 mortar.

Samples arranged from left to right in order of increasing carbon content.

ash are reported as ratios (in percentages) of the strength of the control mortar. By this means, direct comparisons can be made to study the effect of any variable included in these tests. Figures 4 and 5 show averages of the compressive strength ratios obtained with the three cements for each fly ash.

As shown in these tables and figures, an increase in age of test is usually accompanied by an increase in strength ratio. This is assumed to indicate that each fly ash tested here has some pozzolanic properties. The few results which do not follow this general trend are believed to be instances where the effects of pozzolanic action have been overshadowed by other experimental variations.

The replacement of part of the cement by fly ash usually resulted in some loss of strength at an age of 28 days, since at this age sufficient pozzolanic action had not taken place to compensate for the reduction in strength caused by the use of less cement. This early loss in strength became greater as the amount of replacement of cement by fly ash was increased. At greater ages, however, some fly ashes furnished sufficient pozzolanic activity to overcome this early deficiency in strength. The general decrease in strength ratio with increase in the carbon content of the fly ash is also of interest, as well as the individual fly ashes which gave results departing from this trend.

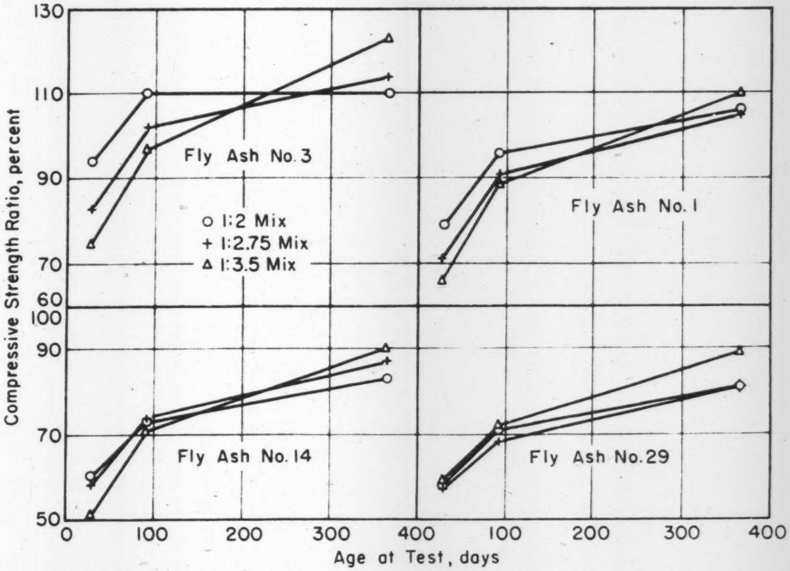


FIG. 6.—Effect of Mix Proportions on Strength of Cement - Fly Ash Mortars.

Averages of values obtained with each of 3 cements with 35 per cent of cement replaced by fly ash.

EFFECT OF RICHNESS OF MIX

As shown in Tables VI, VII, and VIII, four fly ashes, Nos. 1, 3, 14, and 29, were tested in 1:2, 1:2.75, and 1:3.5 mortars. The average results of tests of these mortars obtained with a 35 per cent replacement of the three cements are shown in Fig. 6. For three of the four fly ashes, the richest mix showed the least reduction in strength ratio at an age of 28 days and the leanest mix showed the most. For the fourth fly ash, the strength ratios at 28 days were practically the same. At an age of 1 yr, however, the leanest mix showed the greatest strength ratio for all fly ashes. This shows that ultimately fly ash may be of most value in the leaner mortars and concretes. Similar results have been found with the use of various pozzolans in concrete (5).

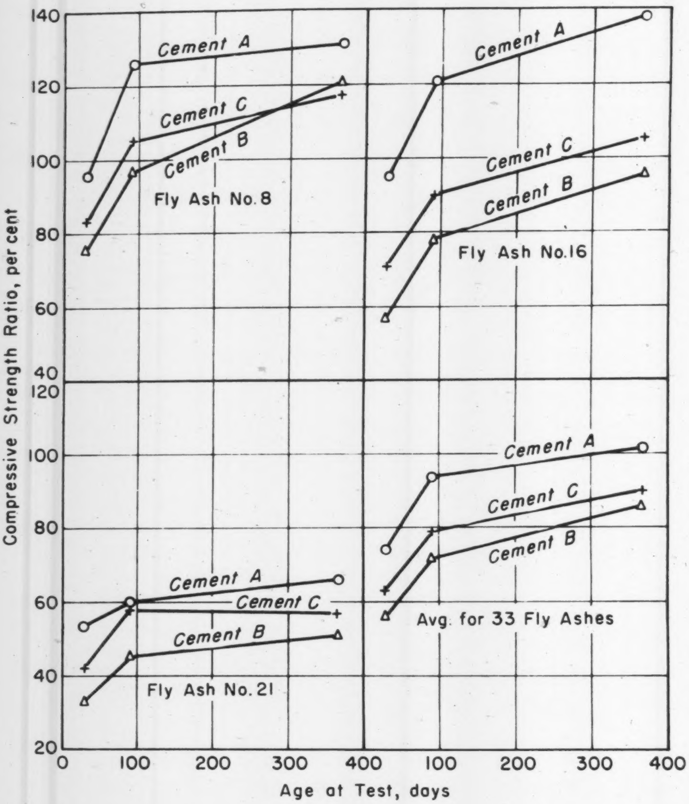


FIG. 7.—Effect of Cement on Strength of Cement - Fly Ash Mortars. 1:2.75 Mortar with 35 per cent Replacement of Cement by Fly Ash.

INFLUENCE OF CHARACTERISTICS OF CEMENT

The characteristics of the cement used had a marked effect on the strength ratio of mortar containing fly ash. The effect of differences in cement is illustrated by the graphs of Fig. 7 which show the age-strength relations obtained for a 35 per cent replacement with each of three fly ashes of different carbon content and also the average of the strength ratios obtained with 33 fly ashes. The use of cement A produced appreciably higher strength ratios than cement B with all fly ashes. With few exceptions, mortars prepared with cement C produced strength ratios intermediate between those shown for mortars containing cement A or B.

To investigate further the variations in strength ratio which might occur because of differences between cements,

TABLE IX.—EFFECT OF CEMENT ON THE COMPRESSIVE STRENGTH RATIO OF CEMENT - FLY ASH MORTAR.

Cement Identification	Properties of Cements				Compressive Strength Ratio of 1:2 Mortars with 50 per cent Replacement of Cement with Fly Ash No. 3, per cent					
	Alkali Content, Equivalent Na_2O, per cent	Tricalcium Silicate Content, per cent	Fineness by Turbidimeter, sq cm per g	Compressive Strength of 1:2 Control Mortar at 28 days, lb per sq in.	3 days	7 days	14 days	28 days	91 days	1 year
M	1.08	27	2100	5025	68	62	81	96	94	83
L	0.82	48	1860	6365	83	87	95	94	97	89
K	0.74	28	2000	5210	61	66	80	89	80	77
J	0.61	52	1800	6600	90	81	85	98	104	100
Average	0.81	39	1940	5800	76	74	85	94	94	87
I	0.50	55	1870	6610	72	67	74	95	103	98
H	0.48	73	2440	8540	69	62	71	85	99	92
G	0.48	39	1760	6415	76	64	72	88	87	90
Average	0.49	56	2025	7190	72	64	72	89	96	93
F	0.38	42	1860	7950	81	68	71	74	92	85
E	0.24	46	2040	7135	69	67	76	85	93	99
D	0.19	61	1780	8125	81	72	76	72	70	84
Average	0.27	50	1890	7740	77	69	74	77	85	89

compressive strength specimens were prepared with 10 cements other than those previously used. In each case, a 1:2 mortar was prepared with fly ash No. 3 replacing 50 per cent of the cement. The cements were selected to provide a wide range in alkali content, tricalcium silicate content, and mortar strength. Cements A, B, and C also varied considerably in these properties. The results obtained with the 10 cements are shown in Table IX. As in the previous tests, a wide range in compressive strength ratios was found for the single fly ash used.

The samples of cement in this table have been separated into three groups on the basis of their alkali content. As shown in Fig. 8 the rate of gain in strength ratio for the groups of mortars prepared with high-, medium-, and low-alkali cements was influenced by the alkali content of the cements. The mortars prepared with high-alkali cement reached maximum strength ratios at some age between 28 and 91 days. Those prepared with medium-alkali cement required about 91 days to gain a maximum strength ratio. The

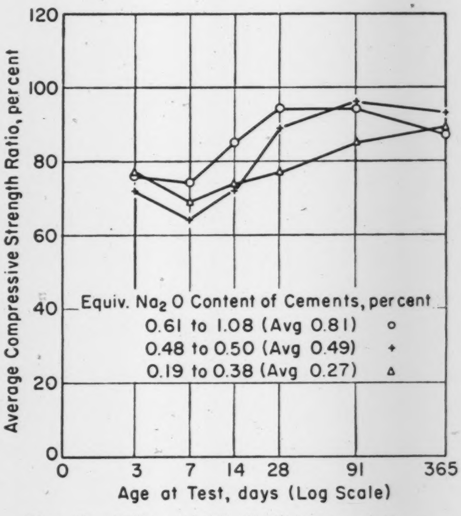


FIG. 8.—Effect of Alkali Content of Cement on Strength of Cement - Fly Ash Mortar (50 per cent Replacement—1:2 Mortar).

mortars prepared with the cements of the low-alkali group appeared to be still gaining in strength ratio when the test was terminated at an age of 1 yr.

Davis (5) has stated that a larger replacement of cement by a pozzolan may be made when type I or II cement is used than when type IV cement is used. Types I and II cements contain more tricalcium silicate than type IV and should liberate more lime which can combine with the pozzolan. To check this hypothesis, the data given in Table IX were rearranged on the basis of the tricalcium silicate content of the cements and placed in groups in which the average tricalcium silicate content of the cement was 31, 45, and 60 per cent. The age-strength data for these groups are shown in Fig. 9. At an age of 1 yr, when the strength resulting from pozzolanic action would be more fully developed for all cements than at early ages, the strength ratios of the three groups of cement vary directly with the tricalcium silicate content. At ages less than 1 yr, no definite relation between strength and tricalcium silicate is apparent.

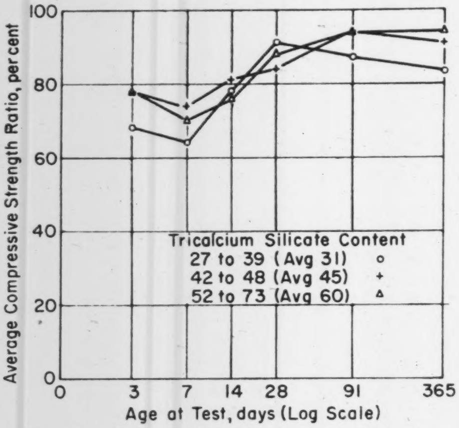


FIG. 9.—Effect of C_3S Content of Cement on Strength of Cement - Fly Ash Mortar (50 per cent Replacement—1:2 Mortar).

The data given in Table IX and shown in Figs. 8 and 9 indicate that the cement used has a marked effect on the development of strength from pozzolanic action in a cement-fly ash mortar. At the earlier ages of test, the alkali in the cement appears to accelerate the reaction between the cement and the fly ash. This agrees with data reported by Alexander (6). At the greater ages of test, the type of cement, or more definitely the amount of tricalcium silicate in the cement, appears to govern the benefits derived by the addition of fly ash to the mortar.

POZZOLANIC STRENGTH INDEX

To determine the relations existing between the strength of cement - fly ash mortars and the properties of fly ash, it was necessary to select one standard of comparison for the strength of the mortar. The strength ratios of mortar obtained at an age of 28 days with 35 per cent replacement of cement by fly ash were considered the most suitable for this purpose. A large percentage of replacement, such as this amount, was found to show the effect of different fly ashes on the strength of mortar to the most marked extent. A 35 per cent replacement was also desirable as it approximated the maximum amount of fly ash generally used in concrete. To obtain the most dependable value for assessing the quality of each fly ash, it appeared desirable to average the results for the three cements used. This average of the strength ratios obtained for each fly ash at an age of 28 days with 35 per cent replacement of each of the three cements will be referred to in the remainder of this report as the "pozzolanic strength index."

The pozzolanic strength indexes of the 33 fly ashes are plotted in Fig. 10

together with averages of the strength ratios obtained on the same mortars at 91 and 365 days. The samples are arranged from left to right in decreasing order of their pozzolanic strength index. If the average strength ratios for 91 or 365 days were used for rating the fly ashes, it can be seen that the order for each age would be somewhat different from that shown. However, the 28-day results should have most significance regarding the use of fly ash in concrete for highways since this is the age at which final strength tests are customarily made and at which the concrete may be placed in service. It should be noted, moreover, that a high pozzolanic strength index generally indicates a continued high level of strength development. To illustrate, the 13 fly ashes having pozzolanic strength indexes of 70 or greater showed an average increase in strength ratio of 33.7 per cent from 28 to 365 days whereas the 20 fly ashes having indexes of less than 70 showed an average increase of only 25.4 per cent during the same period. All of the 13 fly ashes in the group having the higher pozzolanic strength indexes showed increases in strength ratio greater than 25.4 per cent, but only one fly ash of the lower index group had an increase in strength ratio of over 33.7 per cent. It appears that there is a marked difference in quality between the two groups.

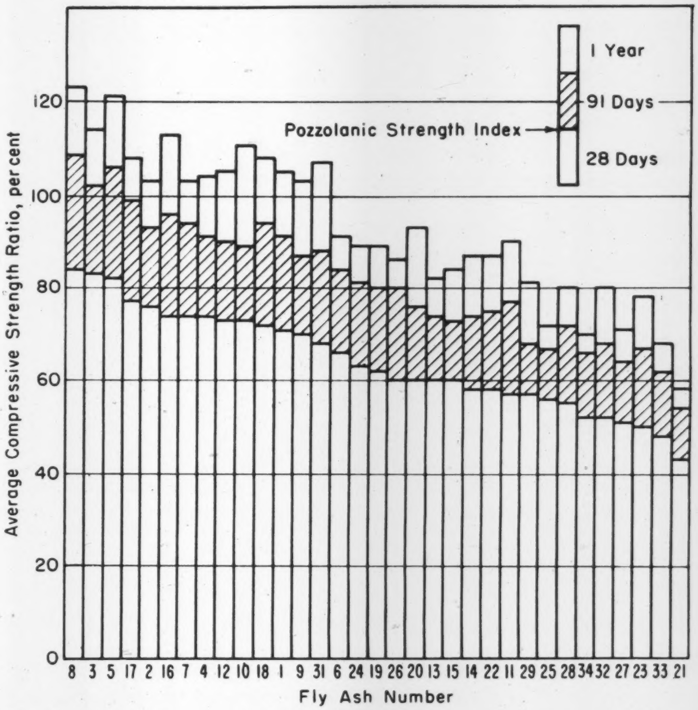


FIG. 10.—Development of Compressive Strength in Cement - Fly Ash Mortar for 33 Fly Ashes.

NOTE.—Values plotted are the average of the compressive strength ratios obtained with a 35 per cent replacement of each of three cements in a 1 :2.75 mortar.

RELATION BETWEEN FINENESS OF FLY ASH AND POZZOLANIC STRENGTH INDEX

A high degree of fineness is generally desired for any pozzolanic material used as an admixture in concrete. The results of the several fineness tests shown in Table II are plotted against the pozzolanic strength index in Fig. 11. No significant relation was found between the specific surface as determined by the air permeability or hydrometer methods and the pozzolanic strength index. Even for fly ashes having low carbon contents (5.0 per cent or less) no definite correlation was found between strength and specific surface. The amount of material passing the No. 325 sieve, however, showed a reasonably definite relation to the pozzolanic strength index. In view of the results obtained with the No. 325 sieve, data from the hydrometer determinations were used to obtain values for amounts of each sample finer than the 0.03-, 0.025-, 0.02-, and 0.01-mm sizes. Correlation with the pozzolanic strength index was found to be best when the size finer than 0.03 mm was used, but became poorer with the smaller sizes in the group.

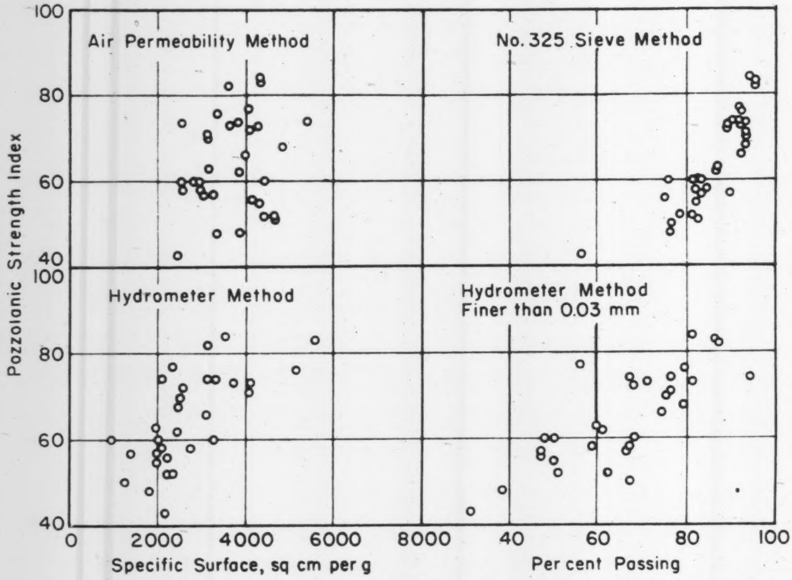


FIG. 11.—Comparison Between Fineness of Fly Ash and Pozzolanic Strength Index.

RELATION BETWEEN CARBON AND THE POZZOLANIC STRENGTH INDEX

Minnick (7) has shown that the strengths of fly ash cement mortars are adversely affected by fly ashes having high carbon contents. In general, the data shown in Tables VI, VII, and VIII support these findings. A similar trend is evident in Fig. 12 in which the relation between the pozzolanic strength index and carbon content is shown. The scattering of points in this figure indicates the effect of variables other than carbon. For example, fly ashes 21 and 31 appear to have properties which cause them to deviate significantly from the general trend. The rather low strength index value of sample 21 may be partly attributed to an unusual coarseness of the fly ash and high water requirement of the mortar prepared with this material. Although sample 31 contained 13.6 per cent carbon, its high fineness and low water requirement probably account for the high strength index.

The effect of carbon content on the strength of mortar was investigated in a special series of tests in which five selected fly ashes having carbon contents ranging from 0.2 to 14.3 per cent were tested before and after heating to a temperature of 550 C. This temperature is considered sufficient to remove all the carbon and, although some oxidation of ferrous iron to the ferric state may have occurred, it is believed that the pozzolanic properties of the inorganic portion of the fly ash were unaltered.

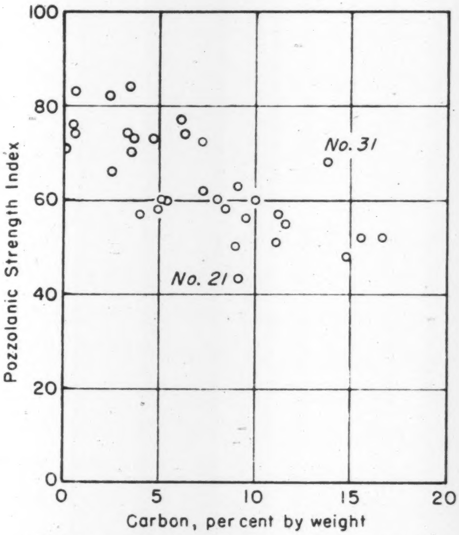


FIG. 12.—Relation Between Carbon Content of Fly Ash and Pozzolanic Strength Index.

Strength tests were conducted on control specimens prepared from a 1:2.75 mortar and test specimens in which a portion of the cement was replaced with fly ash by three different methods as follows:

1. Thirty-five per cent of the solid volume of cement used in the control mortar was replaced by an equal volume of fly ash as received.

2. Thirty-five per cent of the solid volume of cement used in the control mortar was replaced by an equal volume of ignited fly ash. This replacement resulted in more inorganic material being added to the mix than by the first method.

3. Thirty-five per cent of the solid volume of cement used in the control mortar was replaced by a smaller volume of ignited fly ash equal to that of the inorganic portion of the fly ash added by the first method. The replacement in this case did not result in a 1:2.75 mix but one in which the cement, sand, and inorganic constituents from the fly ash were the same as for the mortar in method 1.

The water requirement and strength ratios for this series are shown in Table X. Included with this table is a schematic representation of the three methods of replacement described above. The results obtained with fly ash No. 1 were essentially the same for the three methods of replacement which indicates that the pozzolanic properties of this fly ash were not changed by ignition at 550 C. No effect would be expected to result from the removal of the 0.2 per cent carbon originally present in this sample. An explanation of the strength ratios obtained with fly ash No. 7 is not apparent.

For the remaining three samples, the strength ratios were highest when replacement was made by method 2 which provided the greatest amount of carbon-free fly ash, and lowest by method 1 where carbon was present in the fly ash. The direct effect of the carbon is found

TABLE X.—EFFECT OF CARBON IN FLY ASH ON THE COMPRESSIVE STRENGTH OF CEMENT-FLY ASH MORTAR.

Values are in per cent.

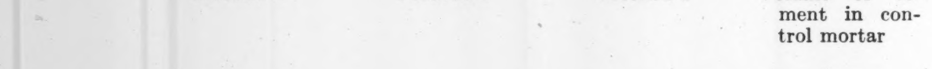
Fly Ash Identification	Method of Replacing Cement with Fly Ash[a]	Carbon Content of Fly Ash	Water Requirement Ratio, Based on Control Mortar	Compressive Strength Ratio, Based on 1:2.75 Control Mortar at	
				28 days	91 days
No. 1	1	0.2	84	89	108
	2	0.0	82	87	108
	3	0.0	83	86	104
No. 7	1	3.5	96	80	99
	2	0.0	86	74	85
	3	0.0	86	76	90
No. 14	1	5.0	100	66	76
	2	0.0	88	71	84
	3	0.0	88	68	83
No. 29	1	11.2	96	65	81
	2	0.0	86	85	106
	3	0.0	86	68	86
No. 32	1	14.3	104	57	69
	2	0.0	90	71	90
	3	0.0	90	61	75

[a] Method 1.—Thirty-five per cent of the solid volume of cement used in the control mortar replaced by an equal volume of fly ash as received.

Method 2.—Thirty-five per cent of the solid volume of cement used in the control mortar replaced by an equal volume of fly ash after ignition at 550 C.

Method 3.—Thirty-five per cent of the solid volume of cement used in the control mortar replaced by an amount of ignited fly ash equal in volume to that of the inorganic portion of the fly ash used in Method 1.

The effect of each method of replacement is illustrated below.



by comparing the results obtained by methods 1 and 3 since the amount of the inorganic constituent is the same in either case. The slightly lower strengths obtained by method 1 where carbon was present can be attributed to the higher water requirements of these mortars. The higher strengths obtained by method 2 as compared to method 3 must result from the greater quantity of carbon-free ash added by that method since no carbon is present in either case. The range in strength ratio at 28 days from 71 to 87 per cent which was found for the five fly ashes tested by method 2 also indicate that essential differences exist between the fly ashes which cannot be attributed to their carbon content.

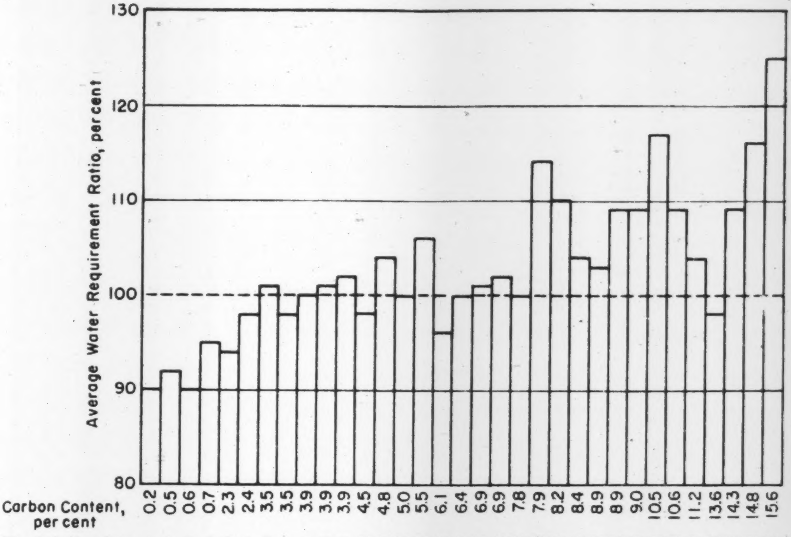


FIG. 13.—Water Requirement Ratios of 33 Fly Ashes.

Note.—Average water requirement ratios are the averages of the ratios obtained with 35 per cent replacement of each of three cements in a 1 : 2.75 mortar.

These results indicate that the carbon in fly ash has at least two adverse effects on the strength of mortar. Some reduction in strength occurs as a result of the increased water requirement of the mortar caused by the presence of carbon. A reduction in strength also appears to result from the fact that the amount of beneficial inorganic material added to the mix is reduced by the amount of carbon present.

RELATION OF WATER REQUIREMENT TO POZZOLANIC STRENGTH INDEX

In the preparation of the mortars for the compressive strength tests, it was found that the amount of water required for uniform consistency of cement-fly ash mortars was not constant. The effect of each fly ash on the water requirement of a 1:2.75 mortar is shown in Fig. 13, where the samples are arranged from left to right in increasing order of their carbon content. As stated before, the water requirement ratio is the amount of water used to prepare a mortar con-

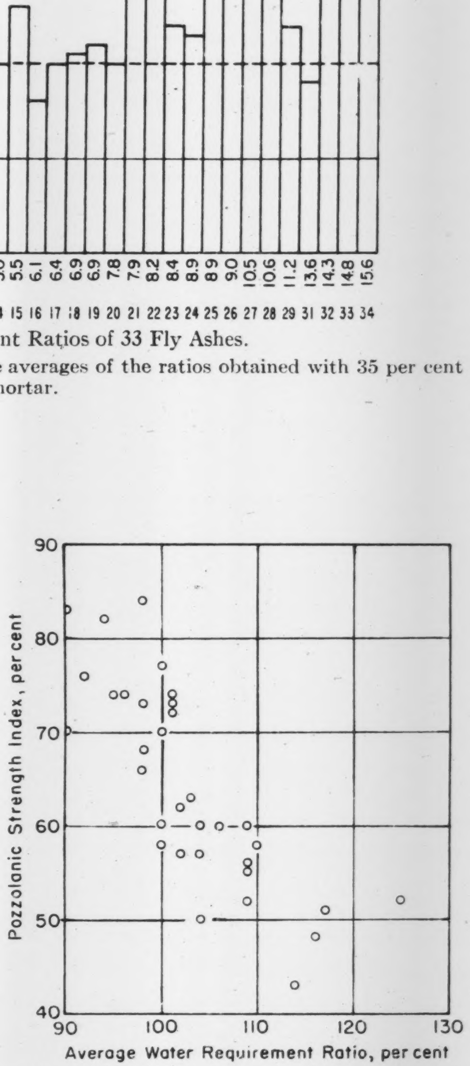


FIG. 14.—Relation Between Water Requirement Strength of Cement - Fly Ash Mortar.

Note: Values based on a 35 per cent replacement of each of three cements with fly ash in a 1 :2.75 mortar.

taining fly ash expressed as a percentage of the amount of water used to prepare the corresponding control mortar. The ratios plotted are average values for the 35 per cent replacement mortars prepared with all three cements. Some fly ashes which contained very little carbon improved the workability of the mortar to such an extent that less water was required for the mortar containing fly ash than for the control mortar. The quantities of water required for mortar of uniform consistency generally increased with the carbon content of the fly ash, although the departures from this trend indicate that differences in physical characteristics also affect the water requirement as would be expected.

The relation between the pozzolanic strength index and the water requirement ratio is shown in Fig. 14 and appears to be somewhat better than that obtained with either carbon content (Fig. 12) or fineness as determined by the No. 325 sieve (Fig. 11). This would appear to be logical, since both the carbon content and fineness of fly ash tend to affect the amount of water needed for a uniform consistency of mortar.

RELATION OF CHEMICAL CONSTITUTION TO POZZOLANIC STRENGTH INDEX

The pozzolanic strength indexes were plotted against the total amounts of each of the inorganic constituents previously given in Table I. These include alumina, iron oxide, calcium oxide, magnesium oxide, sulfur trioxide, alkalis as equivalent sodium oxide, and silica. No indication of a definite relation was found in any of these plots. Even though no relations were found, plots of pozzolanic strength index against total alkalis as Na_2O and silica are shown in Fig. 15 because of the general interest in these constituents.

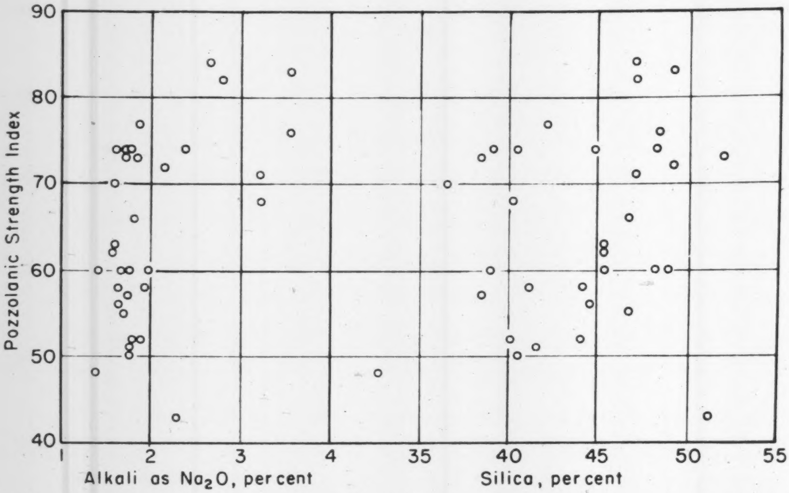


FIG. 15.—Comparison Between Alkali or Silica Content of Fly Ash and Pozzolanic Strength Index.

Considering the general relation found between the alkali content of cement and mortar strength ratios at 28 days (Table IX) it might be thought that the alkalis in the fly ash would be a significant factor. Figure 15 shows that those fly ashes with the highest alkali content had high pozzolanic strength indexes, but for most of the materials the strength index appears to be independent of the alkali content. The silica in fly ash is of interest as it might be expected from the definition of a pozzolanic material that this constituent would have some effect on the strength of mortar. It is possible that silica may influence the strength of mortar to some extent, but the scattered pattern obtained in Fig. 15 shows that there is no direct relation that applies generally to all fly ashes.

TABLE XI.—EFFECT OF FLY ASH ON THE VOLUME CHANGE OF AN ALKALI-REACTIVE MORTAR CONTAINING OPAL.

Fly Ash Identification	Expansion of Mortar Bars Stored at 100 F.[a]									
	Age of 1 Month					Age of 1 Year				
	Amount of Cement Replaced by Fly Ash, per cent									
	0	10	20	35	50	0	10	20	35	50
No. 12	0.02	0.04	0.02	0.00	0.01	0.56	0.35	0.04	0.01	0.02
No. 9	0.14	0.12	0.00	0.01	0.00	0.52	0.43	0.09	0.00	0.00
No. 5	0.12	0.07	0.02	0.00	0.00	0.53	0.39	0.09	0.01	0.01
No. 28	0.06	0.10	0.02	0.00	0.02	0.38	0.37	0.08	0.01	0.01
No. 8	0.06	0.12	0.02	0.01	0.02	0.41	0.40	0.11	0.02	0.02
No. 19	0.10	0.06	0.00	0.00	0.01	0.43	0.45	0.13	0.01	0.02
No. 11	0.09	0.08	0.02	0.02	0.02	0.39	0.40	0.12	0.02	0.01
No. 4	0.10	0.03	0.02	0.00	0.01	0.62	0.54	0.19	0.01	0.00
No. 13	0.05	0.10	0.01	0.00	0.02	0.63	0.48	0.20	0.00	0.02
No. 10	0.02	0.03	0.01	0.00	0.00	0.44	0.45	0.15	0.01	0.00
No. 15	0.08	0.05	0.00	0.00	0.00	0.50	0.47	0.17	0.02	0.01
No. 7	0.11	0.12	0.02	0.01	0.01	0.46	0.46	0.18	0.01	0.01
No. 6	0.07	0.04	0.02	0.02	0.01	0.38	0.30	0.17	0.02	0.01
No. 16	0.02	0.07	0.02	0.01	0.02	0.54	0.55	0.27	0.02	0.03
No. 25	0.04	0.12	0.02	0.02	0.02	0.40	0.54	0.25	0.03	0.02
No. 34	0.09	0.10	0.02	0.01	0.02	0.45	0.49	0.30	0.04	0.01
No. 27	0.05	0.10	0.04	0.02	0.01	0.44	0.46	0.39	0.02	0.01
Average	0.07	0.08	0.02	0.01	0.01	0.48	0.44	0.17	0.02	0.01

[a] Specimens prepared with Cement A (equivalent $Na_2O = 0.90$ per cent). Each value is an average for two specimens.

USE OF FLY ASH TO PREVENT EXPANSION BY ALKALI-AGGREGATE REACTION

One cause of failure in concrete is the expansion and cracking that may result from the attack of certain reactive silica constituents of the aggregate by the alkalis in the cement. It has been found that the destructive effects of this reaction can be prevented by the addition to the concrete of a very finely divided siliceous material which is itself reactive with the alkalis in cement.

The effectiveness of 17 fly ashes in preventing or reducing the expansion resulting from the alkali-aggregate reaction was determined by measuring the change in length of 1 by 1 by $11\frac{1}{4}$-in. mortar bar specimens stored in moist air at 100 F. Control specimens were prepared from a basic reactive mortar consisting of a 1:2 mix by weight using graded Ottawa sand containing 2 per cent of reactive opal passing the No. 8 and retained on the No. 50 sieve. Each fly ash was used to replace 10, 20, 35, or 50 per cent of the cement on a solid volume basis. In all other respects the procedure outlined in the test for Potential Alkali Reactivity of Cement-Aggregate Combinations[12] was followed. All cement-fly ash combinations with opal added were prepared with both cements A and B, which contained 0.90 and 0.13 per cent equivalent Na_2O, respectively. In addition, specimens without opal were prepared with both cements and various percentages of each fly ash.

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[12] Tentative Method of Test for Potential Alkali Reactivity of Cement-Aggregate Combinations (C 227 – 51 T), 1951 Supplement to Book of ASTM Standards, Part 3, p. 13.

All specimens prepared either without opal or with the low-alkali cement showed expansions of less than 0.05 per cent at an age of 1 yr and are not reported here. The expansion of mortars containing opal and prepared with cement A (0.90 per cent alkali) are shown in Table XI for ages of 1 month and 1 yr. All specimens prepared with the same fly ash were made on the same day with a set of control specimens and were stored and tested together. Although the different sets of control specimens failed to develop the same amount of expansion, each set may be used to judge the effect of the single fly ash to which it applies.

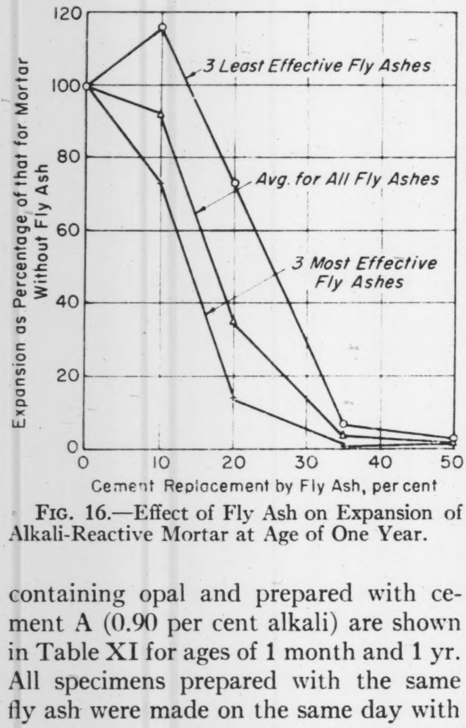

FIG. 16.—Effect of Fly Ash on Expansion of Alkali-Reactive Mortar at Age of One Year.

It is evident that any of the fly ashes tested will prevent the expansion caused by the alkali-aggregate reaction if sufficient quantity is used. At an age of 1 yr, for example, replacement of 35 or 50 per cent of the cement by any of the fly ashes limited the change in volume to a maximum of 0.04 per cent which is comparable with that shown by nonreactive mortars. All specimens have been measured to an age of 2 yr but no significant increase in volume has occurred in those having a replacement of 35 or 50 per cent fly ash. Although a replacement of 20 per cent of the cement by fly ash reduced the expansion at 1 yr it did not reduce it to a safe amount in all cases. The reduction effected by a 20 per cent replacement varied considerably for different fly ashes, indicating that they are not equally effective in preventing the expansion resulting from the alkali-aggregate reaction. This difference in effectiveness is illustrated in Fig. 16.

For the particular reactive mortar used in these tests, a 10 per cent replacement of cement with fly ash did not produce a substantial reduction in expansion and in some cases, the change in volume was increased. At an age of one year, specimens prepared with 7 of the 17 fly ashes in the 10 per cent replacement group showed more expansion than the control specimens.

It should be emphasized that the mortar tests upon which this discussion is based may not reflect the actual behavior of materials when used in concrete. At the present time, tests of mortar bars cannot be relied upon as substitutes for tests of concrete specimens to determine the amount of a particular fly ash which must be used to prevent expansion in a reactive concrete.

SPECIAL CHEMICAL STUDIES

Since strength tests of mortar are time consuming, there has been considerable interest in the development of more rapid tests to measure the activity of pozzolans. Most of these tests

TABLE XII.—ALKALINITY AND COMPOSITION OF WATER EXTRACTS OF FLY ASH.

Values are in per cent (except pH)

Identification	pH of Extract	Calculated Composition of Dissolved Material[a]				Total Dissolved Material[a]		
		$CaSO_4 \cdot 2H_2O$	$Ca(OH)_2$	NaOH	KOH	Calculated	Determined (Dried at 110 C)	Difference (Determined Less Calculated)
No. 1	11.8	3.66	1.22	0.23	0.06	5.17	5.16	−0.01
No. 2	11.7	3.02	0.65	0.34	0.07	4.08	4.08	0.00
No. 3	11.7	4.41	0.18	0.45	0.11	5.15	5.19	0.04
No. 4	11.8	2.02	1.43	0.03	0.06	3.54	3.49	−0.05
No. 5	11.8	2.87	1.00	0.18	0.06	4.11	3.93	−0.18
No. 6	11.8	3.12	1.44	0.06	0.06	4.68	4.55	−0.13
No. 7	11.0	2.18	0.13	0.05	0.10	2.46	2.54	0.08
No. 8	11.7	1.75	0.18	0.08	0.06	2.07	2.14	0.07
No. 9	12.0	1.59	2.02	0.05	0.02	3.68	4.48	0.80
No. 10	12.3	2.97	3.80	0.04	0.05	6.86	6.65	−0.21
No. 11	10.6	0.95	0.15	0.03	0.02	1.15	1.30	0.15
No. 12	11.3	0.79	0.33	0.03	0.02	1.17	1.30	0.13
No. 13	10.1	0.47	0.04	0.01	0.02	0.54	0.73	0.19
No. 14	12.0	1.13	1.25	0.08	0.02	2.48	2.94	0.46
No. 15	11.9	1.29	1.66	0.04	0.02	3.01	3.15	0.14
No. 16	12.3	2.68	4.48	0.04	0.02	7.22	7.15	−0.07
No. 17	11.4	1.84	0.42	0.05	0.02	2.33	2.28	−0.05
No. 18	11.3	0.97	0.30	0.04	0.05	1.36	1.58	0.22
No. 19	9.9	0.64	0.03	0.03	0.04	0.74	0.77	0.03
No. 20	12.1	0.40	2.59	0.01	0.02	3.02	3.92	0.90
No. 21	11.3	0.47	0.46	0.03	0.04	1.00	1.22	0.22
No. 22	12.3	1.46	3.74	0.01	0.02	5.23	5.31	0.08
No. 23	12.0	2.42	2.92	0.04	0.02	5.40	5.34	−0.06
No. 24	9.0	0.65	0.00	0.04	0.00	0.69	0.70	0.01
No. 25	10.4	1.54	0.17	0.01	0.02	1.74	1.78	0.04
No. 26	8.5	0.73	0.00	0.04	0.05	0.82	0.78	−0.04
No. 27	12.0	3.19	2.30	0.06	0.06	5.61	5.45	−0.16
No. 28	9.3	0.64	0.00	0.03	0.02	0.69	0.75	0.06
No. 29	11.1	1.04	0.41	0.03	0.01	1.49	1.62	0.13
No. 30	11.9	0.89	0.45	0.08	0.02	1.44	1.67	0.23
No. 31	11.0	4.35	0.07	0.34	0.22	4.98	4.80	−0.18
No. 32	10.8	0.43	0.12	0.03	0.02	0.60	0.68	0.08
No. 33	10.0	2.81	0.17	0.03	0.06	3.07	3.12	0.05
No. 34	11.2	3.03	0.65	0.03	0.00	3.71	3.84	0.13

[a] Expressed as a percentage of the original weight of fly ash.

have attempted to measure in some way special chemical properties of the pozzolanic materials alone or the chemical changes occurring in mixtures of lime and pozzolans. In their paper on determining pozzolanic activity, Moran and Gilliland (8) give a summary of most of these tests. They point out in summation that "a single short-time test will not evaluate pozzolanic activity, particularly when any one of several properties may be desired in a given material. It appears that each material requires rather exhaustive testing, after which it may be possible to empirically relate a quick test for control purposes." The special chemical tests made in this investigation were conducted with this latter objective in mind. They were undertaken to supply a background of information for further work so that ultimately it might be possible to establish interrelations between various properties and test results which would make possible more rapid methods of test.

TABLE XIII.—THE DISTRIBUTION OF CALCIUM IN FLY ASH.
Values are in per cent.

Identification	Calcium as CaO from[a]				
	Total Sample	Total Water-Soluble	Water-Soluble $CaSO_4$	Water-Soluble $Ca(OH)_2$	Free[b] Lime
No. 1	7.0	2.1	1.2	0.9	0.9
No. 2	5.2	1.5	1.0	0.5	0.5
No. 3	5.5	1.6	1.4	0.2	0.0
No. 4	5.2	1.7	0.7	1.0	0.9
No. 5	5.2	1.7	0.9	0.8	0.6
No. 6	6.4	2.1	1.0	1.1	0.8
No. 7	1.7	0.8	0.7	0.1	0.2
No. 8	4.5	0.7	0.6	0.1	0.5
No. 9	7.8	2.0	0.5	1.5	1.6
No. 10	8.3	3.9	1.0	2.9	2.3
No. 11	1.8	0.4	0.3	0.1	0.0
No. 12	2.3	0.5	0.3	0.2	0.1
No. 13	1.6	0.2	0.2	0.0	0.0
No. 14	6.0	1.3	0.4	0.9	1.4
No. 15	6.4	1.7	0.4	1.3	1.0
No. 16	11.6	4.3	0.9	3.4	2.6
No. 17	4.3	0.9	0.6	0.3	0.2
No. 18	3.6	0.5	0.3	0.2	0.2
No. 19	1.4	0.2	0.2	0.0	0.0
No. 20	5.2	2.1	0.1	2.0	1.5
No. 21	1.6	0.5	0.1	0.4	0.3
No. 22	7.6	3.3	0.5	2.8	2.3
No. 23	8.5	3.0	0.8	2.2	1.6
No. 24	1.5	0.2	0.2	0.0	0.0
No. 25	6.2	0.6	0.5	0.1	0.0
No. 26	1.2	0.2	0.2	0.0	0.0
No. 27	6.8	2.8	1.0	1.8	1.0
No. 28	1.1	0.2	0.2	0.0	0.0
No. 29	3.2	0.6	0.3	0.3	0.1
No. 30	12.0	0.6	0.3	0.3	0.9
No. 31	3.9	1.5	1.4	0.1	0.0
No. 32	1.7	0.2	0.1	0.1	0.0
No. 33	1.8	1.0	0.9	0.1	0.0
No. 34	3.5	1.5	1.0	0.5	0.0

[a] Expressed as a percentage of the original weight of fly ash.

[b] Determined by ASTM Method C 114 – 53.

The special tests conducted on the fly ash itself involved chiefly the determination of constituents soluble in water or sodium hydroxide and the changes in solubility resulting from reactions of lime and fly ash in the presence of water.

Table XII shows the amount and calculated composition of the water-soluble material in each of the fly ashes. These data were obtained by shaking a 5-g sample of fly ash in 100 ml of distilled water for 1 hr, allowing the mixture to stand 24 hr and filtering the supernatant liquid without further dilution or washing. The pH of this extract is also recorded in Table XII.

Chemical analyses were conducted on aliquots for the amounts of calcium, sodium, potassium, and sulfate. The total amount of dissolved material was also determined by evaporating the water from an aliquot and drying the residue at 110 C. The composition of the dissolved salts was calculated by assuming that all of the sulfate was combined as gypsum ($CaSO_4 \cdot 2H_2O$). The balance of the calcium was then calculated as calcium hydroxide ($Ca(OH)_2$). The sodium and potassium were assumed to be present as the hydroxides (NaOH or KOH). Generally there is good agreement between the total amount of soluble material and the total amount of calculated salts. This is an indication of the validity of the assumptions made in calculating the composition.

The pH values of the extracts depend on the proportions of water and sample used in the test as well as the composition of the dissolved material, and there is no direct relation between the quantitative amount of any particular constituent and the pH. Generally, the values obtained are those to be expected from a consideration of the calculated amount of calcium hydroxide present. In Table XII, the pH value is relatively low for those extracts having no calcium hydroxide by calculation and high for those extracts in which the calculated calcium hydroxide is large. For all samples that have a calculated lime content greater than 2.0 per cent, the pH of the extract was 12.0 or more.

The data for the soluble salts in Table XII show that essentially all of the extracted water-soluble material is either calcium sulfate or calcium hydroxide, the relative amounts of each varying greatly for different fly ashes. These data, however, do not indicate the state in which the soluble materials are present in the original fly ash. The calcium sulfate may be present in any of its three forms—anhydrous, hemihydrate, or dihydrate—depending upon the conditions surrounding the fly ash during storage or handling. Likewise the calcium hydroxide may be present as uncombined lime or may have formed from the hydrolysis of lime-silica complexes after the addition of water.

To verify the presence of uncombined lime in the fly ash prior to wetting with water, determinations were made by the alcohol-glycerol method, such as is used in the determination of free lime in portland cement.[13] The results of these tests are given in Table XIII. Table XIII also includes the total amount of calcium that is present in the sample and the amount that is water soluble. The part of the water-soluble calcium that is calcium hydroxide and the part that is calcium sulfate are also given. All these results are expressed in terms of an equivalent amount of calcium oxide to provide a common basis of comparison. It can be seen that for most of the samples the determined free lime is approximately the same as the calculated water-soluble calcium hydroxide, thus indicating that the addition of water to fly ash does not result in the formation of calcium hydroxide by the hydrolysis of lime complexes. It will also be noted that a considerable portion of the calcium is not soluble in water

TABLE XIV.—REACTION OF FLY ASH WITH SODIUM HYDROXIDE SOLUTION.[a]
Values are in per cent.

Fly Ash Identification	Reduction in Alkalinity of Solution	SiO_2[b] Dissolved	Al_2O Dissolved	SO_3[b] Dissolved
No. 1	42.6	0.45	0.10	2.38
No. 2	36.7	0.68	0.10	1.92
No. 3	42.1	0.49	0.10	2.48
No. 4	33.8	0.58	0.11	1.33
No. 5	37.2	0.56	0.11	1.66
No. 6	46.1	0.71	0.06	1.92
No. 7	29.3	1.39	0.07	0.93
No. 8	30.0	0.50	0.14	1.10
No. 9	37.5	0.36	0.17	1.21
No. 10	45.2	0.43	0.10	2.09
No. 11	28.7	2.59	0.06	0.47
No. 12	27.3	0.77	0.13	0.38
No. 13	22.5	1.74	0.08	0.22
No. 14	29.0	0.51	0.16	0.80
No. 15	33.8	0.76	0.09	0.91
No. 16	38.1	0.26	0.41	1.58
No. 17	38.7	0.27	0.25	0.95
No. 18	23.9	0.29	0.34	0.49
No. 19	24.2	2.47	0.06	0.32
No. 20	25.0	0.72	0.12	0.27
No. 21	22.2	2.26	0.06	0.22
No. 22	35.7	0.84	0.08	0.86
No. 23	44.3	0.76	0.06	1.54
No. 24	18.2	0.58	0.19	0.30
No. 25	41.4	0.96	0.05	0.76
No. 26	27.9	3.26	0.05	0.35
No. 27	50.7	0.66	0.04	2.06
No. 28	31.5	3.32	0.05	0.35
No. 29	25.8	0.48	0.20	0.50
No. 30	28.7	1.04	0.08	0.70
No. 31	31.8	0.34	0.19	2.02
No. 32	18.2	0.83	0.14	0.17
No. 33	34.1	1.52	0.06	1.31
No. 34	44.9	1.33	0.03	1.50

[a] Fly ash treated with 1 *N* solution of sodium hydroxide for 24 hr at 80 C in a sealed container. ASTM Method C 289 - 52 T, except that a 12.5 g sample was used.

[b] Expressed as a percentage of the original weight of fly ash.

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[13] Methods of Chemical Analysis of Portland Cement (C 114 - 53), 1955 Book of ASTM Standards, Part 3, p. 69.

and, therefore, is probably present as a calcium glass.

The data in Table XIV were obtained by making the test for reactivity of pozzolans with sodium hydroxide solution as described in Appendix A of the report by Moran and Gilliland on determining pozzolanic activity (8). This test is an adaptation of a procedure now designated as the ASTM Tentative Method of Test for Potential Reactivity of Aggregates (Chemical Method).[14] It involves treatment of the sample with 1 *N* solution of sodium hydroxide in a sealed container at 80 C for a 24-hr period. At the end of this period, the solution is filtered and the resulting filtrate examined. In the tests described by Moran and Gilliland only the reduction in alkalinity (the amount of sodium hydroxide consumed by the reactions taking place) and the amount of silica dissolved were determined. In this investigation the amounts of alumina and sulfate dissolved were also determined.

This test was originally proposed for use on calcined shales. For this type of material it showed some promise in evaluating the ability of the pozzolan to prevent expansion resulting from the alkali-aggregate reaction. Consequently, consideration has been given to its application to fly ash. It was found however that the presence of calcium sulfate in a fly ash affects the results obtained by this test. When calcium sulfate dissolves, the calcium ion precipitates much of the silica which might normally be soluble and remain in solution if no calcium were present. The presence of dissolved calcium sulfate might also affect the reduction in alkalinity by causing a precipitation of calcium hydroxide from solution. The data in Table XIII illustrate these effects. In most cases where the calcium sulfate dissolved is relatively high (as indicated by the amount of SO_3), the amount of silica dissolved is low and the reduction in alkalinity is relatively high. It appears that the relation between soluble silica and reduction in alkalinity is controlled by the amount of calcium sulfate present as well as by the characteristics of the siliceous material. Values for reduction in alkalinity and amount of silica dissolved which have been found suitable for acceptance requirements of other types of pozzolans may not be applicable to fly ashes.

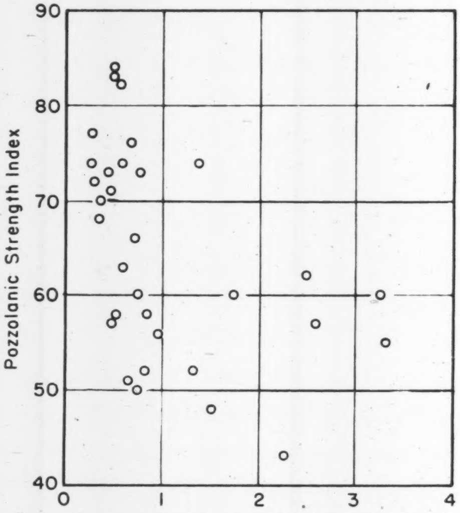

FIG. 17.—Comparison Between Silica Dissolved in NaOH Solution and Pozzolanic Strength Index.

A plot of the pozzolanic strength index against the reduction in alkalinity failed to show any correlation. An apparent relation between the strength developed and the silica dissolved is shown in Fig. 17, but this relation is the inverse of what might normally be expected, that is, that larger amounts of dissolved silica would result in greater pozzolanic activity. As previously stated, the presence

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[14] Tentative Method of Test for Potential Reactivity of Aggregates (Chemical Method) (C 289 – 54 T) 1955 Book of ASTM Standards, Part 3, p. 1206.

TABLE XV.—SOLUBILITY OF ALKALIES IN WATER AFTER VARIOUS PERIODS OF AGING FLY ASH AND LIME SLURRIES.

Values are in per cent.

Identification	Sodium Oxide			Potassium Oxide			Equivalent Alkalies as Sodium Oxide				
	Proportion Soluble at[a]			Proportion Soluble at[a]			Proportion Soluble at[a]			Total in Fly Ash[b]	Soluble at 28 days[b]
	7 days	28 days	91 days	7 days	28 days	91 days	7 days	28 days	91 days		
No. 1	18	42	42	12	34	40	15	38	41	3.21	1.23
No. 2	19	35	46	12	30	42	16	33	44	3.55	1.16
No. 3	25	43	41	13	33	37	20	39	39	3.55	1.38
No. 4	14	29	31	12	37	57	12	35	52	1.61	0.56
No. 5	19	42	45	12	39	47	15	40	46	2.79	1.13
No. 6	22	44	50	18	49	66	19	47	62	1.82	0.86
No. 7	20	40	40	14	48	59	15	46	55	1.78	0.82
No. 8	13	31	37	10	34	41	11	33	39	2.64	0.88
No. 9	15	45	55	10	42	61	12	44	58	1.59	0.70
No. 10	20	42	45	19	52	64	19	49	59	1.72	0.85
No. 11	12	42	35	10	48	52	10	46	48	1.72	0.80
No. 12	12	30	40	9	33	45	10	32	44	1.85	0.60
No. 13	14	36	59	9	41	54	10	40	55	1.40	0.56
No. 14	13	38	47	9	38	45	11	38	46	1.93	0.74
No. 15	13	45	68	10	45	70	12	45	69	1.67	0.75
No. 16	13	38	43	9	39	45	11	39	45	2.38	0.92
No. 17	12	24	48	15	44	53	14	37	51	1.87	0.69
No. 18	14	28	45	12	34	50	12	32	48	2.15	0.69
No. 19	8	36	56	9	46	66	9	44	64	1.57	0.69
No. 20	7	27	54	7	32	49	7	31	50	1.78	0.55
No. 21	8	27	37	8	37	46	8	35	44	2.29	0.79
No. 22	25	28	50	10	43	59	13	40	57	1.62	0.64
No. 23	13	33	60	9	36	65	11	35	63	1.77	0.62
No. 24	13	30	42	10	34	46	11	32	44	1.60	0.52
No. 25	16	34	47	20	47	56	19	45	54	1.64	0.74
No. 26	12	33	52	9	42	64	10	40	60	1.97	0.78
No. 27	21	48	48	22	60	63	22	56	59	1.76	0.98
No. 28	12	36	50	10	44	57	11	52	55	1.85	0.97
No. 29	30	38	62	7	25	39	15	29	47	1.84	0.53
No. 30	18	37	37	16	44	38	17	38	37	1.94	0.74
No. 31	24	46	55	12	37	48	16	41	50	3.11	1.26
No. 32	9	34	41	8	36	48	8	35	46	1.79	0.63
No. 33	20	48	64	12	51	63	14	50	63	1.39	0.70
No. 34	14	46	54	13	51	59	13	49	58	1.88	0.92

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[a] Expressed as a percentage of the amount of the constituent present in the original fly ash sample (Table I).

[b] Expressed as a percentage of the original weight of fly ash.

TABLE XVI.—SILICA DISSOLVED FROM FLY ASH AFTER VARIOUS PERIODS OF AGING FLY ASH AND LIME SLURRIES.

Values are in per cent.

Identification	Acid Soluble at[a]			Alkali-Acid Soluble at[b]		
	7 days	28 days	91 days	7 days	28 days	91 days
No. 1	0.44	0.74	0.44	1.68	2.40	...
No. 2	0.41	0.57	0.41	2.03	1.56	...
No. 3	0.41	0.73	0.45	1.75	2.71	...
No. 4	0.29	0.60	0.35	0.97	1.54	...
No. 5	0.37	0.62	0.38	1.77	1.60	...
No. 6	0.38	0.63	0.40	0.71	1.93	...
No. 7	0.35	0.55	0.34	1.21	1.71	...
No. 8	0.32	0.77	0.39	2.07	2.38	...
No. 9	0.85	0.47	0.47	1.84	2.45	...
No. 10	0.40	0.58	0.44	1.38	1.69	...
No. 11	0.36	0.68	0.42	1.25	1.86	...
No. 12	0.41	0.70	1.07	0.72	2.59	0.92
No. 13	0.38	0.67	1.03	0.91	1.41	0.97
No. 14	0.40	0.73	0.97	1.50	2.15	1.18
No. 15	0.40	0.79	1.00	1.54	2.64	0.95
No. 16	0.42	0.64	0.99	2.06	2.60	1.33
No. 17	0.39	0.75	1.01	1.80	2.73	1.27
No. 18	0.41	0.39	0.64	1.78	2.57	1.14
No. 19	0.37	0.30	0.57	0.74	2.46	1.31
No. 20	0.37	0.34	0.49	0.87	2.26	1.10
No. 21	0.40	0.30	0.59	1.00	2.22	1.76
No. 22	0.34	0.33	...	1.00	1.24	...
No. 23	0.40	0.36	0.63	0.90	1.45	1.87
No. 24	0.44	0.30	0.57	1.11	1.32	0.88
No. 25	0.44	0.32	0.53	1.53	1.85	1.32
No. 26	0.43	0.33	0.50	1.41	2.17	1.32
No. 27	0.38	0.33	0.53	1.38	1.97	1.19
No. 28	0.42	0.32	0.51	1.04	2.16	1.49
No. 29	0.44	0.42	0.51	1.63	1.96	0.95
No. 30	0.46	0.38	0.42	1.73	1.82	1.25
No. 31	0.38	0.29	0.44	1.85	2.16	1.54
No. 32	0.46	0.32	0.49	0.96	1.25	0.98
No. 33	0.42	0.30	0.48	0.85	1.53	0.80
No. 34	0.41	0.36	0.43	1.08	2.05	1.11

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[a] Amount dissolved by digesting the residue from water extraction in HCl (1:10) overnight. Expressed as a percentage of the original weight of fly ash.

[b] Amount dissolved by digesting the residue from the acid treatment for 30 min on steam bath in 1 per cent NaOH solution. HCl added to make solution acid immediately before filtering. Results are expressed as percentages of the original weight of fly ash.

TABLE XVII.—ALUMINA DISSOLVED FROM FLY ASH AFTER VARIOUS PERIODS OF AGING FLY ASH AND LIME SLURRIES.

Values are in per cent.

Identification	Acid Soluble at[a]			Alkali-Acid Soluble at[b]		
	7 days	28 days	91 days	7 days	28 days	91 days
No. 1	3.8	7.2	8.4	0.33	0.38	...
No. 2	3.9	7.0	8.7	0.36	0.36	...
No. 3	3.9	7.8	9.4	0.48	0.53	...
No. 4	2.2	5.6	7.9	0.46	0.31	...
No. 5	3.6	7.9	9.2	0.38	0.32	...
No. 6	3.7	5.9	7.1	0.19	0.37	...
No. 7	2.0	5.7	6.9	0.28	0.36	...
No. 8	4.1	8.5	10.1	0.51	0.62	...
No. 9	4.9	8.2	10.4	0.44	0.42	...
No. 10	2.9	6.2	7.8	0.29	0.29	...
No. 11	2.2	5.8	7.7	0.26	0.35	...
No. 12	3.0	7.1	9.9	0.32	0.38	0.24
No. 13	2.2	5.6	7.7	0.19	0.38	0.25
No. 14	4.1	7.5	9.5	0.30	0.36	0.27
No. 15	3.8	6.8	9.2	0.19	0.31	0.21
No. 16	5.2	9.4	11.0	0.43	0.44	0.38
No. 17	4.9	9.5	12.3	0.51	0.47	0.36
No. 18	4.9	9.9	13.0	0.45	0.53	0.36
No. 19	1.9	5.4	7.4	0.12	0.32	0.23
No. 20	1.9	5.1	8.2	0.15	0.25	0.16
No. 21	1.5	4.6	6.5	0.18	0.36	0.32
No. 22	2.1	5.3	...	0.20	0.16	...
No. 23	2.1	4.8	7.4	0.16	0.21	0.27
No. 24	3.2	6.9	9.9	0.36	0.23	0.25
No. 25	3.7	6.1	7.6	0.38	0.31	0.29
No. 26	2.0	5.3	7.3	0.35	0.32	0.31
No. 27	2.7	6.0	7.2	0.35	0.28	0.26
No. 28	2.0	5.4	7.4	0.20	0.28	0.31
No. 29	4.0	7.5	11.2	0.44	0.44	0.32
No. 30	3.6	5.0	5.5	0.15	0.15	0.20
No. 31	3.6	7.2	9.5	0.45	0.41	0.38
No. 32	2.3	5.7	8.2	0.19	0.19	0.21
No. 33	2.0	5.3	7.8	0.21	0.21	0.11
No. 34	3.0	6.2	7.9	0.32	0.27	0.19

[a] Amount dissolved by digesting the residue from water extraction in HCl (1:10) overnight. Expressed as a percentage of the original weight of fly ash.

[b] Amount dissolved by digesting the residue from the acid treatment for 30 min on steam bath in 1 per cent NaOH solution. HCl added to make solution acid immediately before filtering. Results are expressed as percentages of the original weight of fly ash.

TABLE XVIII.—IRON OXIDE DISSOLVED FROM FLY ASH AFTER VARIOUS PERIODS OF AGING FLY ASH AND LIME SLURRIES.

Values are in per cent.

Identification	Acid Soluble at[a]			Alkali-Acid Soluble at[b]		
	7 days	28 days	91 days	7 days	28 days	91 days
No. 1	1.5	2.4	2.9	0.09	0.09	...
No. 2	1.6	2.5	3.2	0.08	0.12	...
No. 3	2.1	3.1	3.7	0.13	0.16	...
No. 4	1.2	2.1	2.6	0.05	0.10	...
No. 5	1.5	2.8	3.6	0.10	0.09	...
No. 6	0.9	1.6	1.9	0.04	0.10	...
No. 7	0.8	1.7	2.3	0.06	0.12	...
No. 8	1.8	3.1	3.9	0.15	0.23	...
No. 9	1.8	2.3	3.2	0.10	0.17	...
No. 10	1.1	2.0	2.7	0.07	0.10	...
No. 11	0.7	1.6	2.3	0.06	0.09	...
No. 12	0.8	1.7	2.4	0.08	0.10	0.08
No. 13	0.6	1.3	2.0	0.07	0.10	0.13
No. 14	1.3	1.7	2.8	0.07	0.12	0.17
No. 15	1.1	1.6	2.6	0.04	0.10	0.15
No. 16	2.2	3.2	3.9	0.09	0.16	0.21
No. 17	2.0	3.2	4.5	0.16	0.16	0.17
No. 18	1.4	1.9	2.5	0.07	0.10	0.08
No. 19	0.7	1.6	2.3	0.06	0.10	0.14
No. 20	1.4	2.7	4.3	0.09	0.14	0.12
No. 21	0.6	1.2	1.5	0.06	0.09	0.08
No. 22	1.4	2.5	...	0.10	0.08	...
No. 23	1.0	1.6	2.1	0.06	0.06	0.37
No. 24	0.8	1.7	2.4	0.09	0.07	0.11
No. 25	1.3	2.1	2.8	0.12	0.12	0.15
No. 26	0.7	1.7	2.7	0.09	0.08	0.15
No. 27	1.0	1.8	2.1	0.15	0.12	0.17
No. 28	0.6	1.5	2.1	0.07	0.07	0.15
No. 29	1.0	1.9	2.0	0.09	0.10	0.12
No. 30	1.9	2.1	2.3	0.06	0.07	0.11
No. 31	1.8	2.9	3.9	0.12	0.16	0.24
No. 32	0.7	1.6	3.8	0.07	0.08	0.14
No. 33	1.0	2.0	2.4	0.15	0.17	0.20
No. 34	1.0	1.8	2.6	0.17	0.21	0.21

[a] Amount dissolved by digesting the residue from water extraction in HCl (1:10) overnight. Expressed as a percentage of the original weight of fly ash.

[b] Amount dissolved by digesting the residue from the acid treatment for 30 min on steam bath in 1 per cent NaOH solution. HCl added to make solution acid immediately before filtering. Results are expressed as percentages of the original weight of fly ash.

of calcium sulfate controls to a considerable extent the amount of silica remaining in solution. For this reason no particular significance can be attached to this apparent relation.

The amount of Al_2O_3 dissolved varied from 0.03 to 0.41 per cent, the median being 0.1 per cent. No significance could be attached to the variations in these values.

The data given in Tables XV through XVIII were obtained by analysis of a slurry of fly ash, lime, and water that had been stored in an oven at 100 F for periods of 7, 28, and 91 days. The procedure used for the preparation and storage of the slurries was essentially the same as that given for the determination of "available alkalis" in the Tentative Methods of Sampling and Testing Fly Ash for Use as an Admixture in Portland-Cement Concrete.[15] This test has been proposed as a relative measure of the amounts of alkalis from the fly ash that are made available for reaction with any reactive aggregate that may be present in the concrete. For this study tests were made on slurries at ages of 7 and 91 days as well as the 28 days called for in the ASTM method. Some variation in technique from the ASTM method was found necessary to insure better control of test conditions, and the details of the test procedure used for this investigation are given in Appendix I (Procedure 2).

Table XV shows the individual amounts of sodium and potassium oxides that were extracted with water after each test period and the total of these two expressed as an equivalent amount of sodium oxide. Each result is expressed as a percentage of the total amount of the constituent present in each fly ash. The total amounts of alkalis present in each sample and the alkalis soluble at 28 days expressed as percentages of the original weight of fly ash are also given. These results invariably show considerable increase in the solubility of alkali between 7 and 28 days, but the results for the 91-day period differ with the samples tested. Some show little or no further increase but others show a marked increase in the alkali dissolved. This increase was as much as 28 per cent of the equivalent (total) alkali in the case of sample 23. The increase was greater than 10 per cent in 18 of the 34 samples. It is evident that storage of lime - fly ash slurry for 28 days does not yield a result for soluble alkalis that can be related to the ultimate solubility of the alkalis in lime solution.

In order to obtain additional information of the chemical changes taking place in intimate mixtures of fly ash and lime in the presence of water, the residues after the extraction of the alkalis were digested in dilute hydrochloric acid (1:10). The acid solution was filtered off and the residue from this treatment digested in sodium hydroxide (1 per cent). This was then filtered. Each of the filtrates thus obtained were examined for the amounts of silica, alumina, and iron oxide present. The details of the procedure used are described in Appendix I (Procedure 2). These results are shown in Tables XVI, XVII, and XVIII, respectively.

Because of the exploratory nature of these tests, relatively large experimental errors are likely, but the results are of interest because they show that the solubility of the alumina (Table XVII) was increased to a much greater extent during the period covered by the tests than was the solubility of the silica (Table XVI). Table XVI shows that for some samples there is a general trend toward larger amounts of acid-soluble silica being present for the longer period of storage, but the results are erratic. In all cases only a small fraction of the total amount of silica present was soluble in acid. The change with age in the acid solubility of the alumina shown in Table XVII is considerable. The acid-soluble alumina at 7 days is in the range of 2 to 4 per cent for most samples, but at 91 days this range is 7 to 10 per cent. Generally, the increase in the solubility of the alumina between 7 and 91 days represented from 20 to 30 per cent of the total alumina in the sample. The solubility of the iron oxide shown in Table XVIII shows somewhat the same trend as shown by that of the alumina but to a lesser degree.

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[15] Tentative Methods of Sampling and Testing Fly Ash for Use as an Admixture in Portland Cement Concrete (C 311 – 54 T), 1955 Book of ASTM Standards, Part 3, p. 1332.

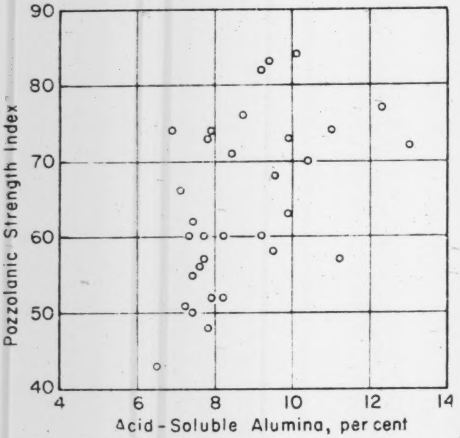

FIG. 18.—Comparison Between Acid-Soluble Alumina After Storage in Lime Solution for 91 Days and Pozzolanic Strength Index.

Because of the considerable increase with time in the solubility of alumina in the presence of lime and water, the relation of this acid-soluble alumina to the pozzolanic strength index is of interest (Fig. 18). There is a general trend toward higher strength ratios for larger amounts of acid-soluble alumina, but it is also apparent that samples having widely different amounts of acid-soluble alumina have essentially the same strength index. Although the reactivity of the alumina may be a factor in the development of strength, other factors must exert a greater influence.

The amounts of alkali-soluble material shown in Tables XVI, XVII, and XVIII have little significance. This alkali digestion was made primarily to determine whether significant changes occurred in the amount of amorphous silica present in the fly ash during reaction with lime. The test procedure was based on the assumption that only this form of silica would be insoluble in the acid treatment and soluble in the alkali solution. However, the glassy constituents of the fly ash were evidently decomposed to some extent by the conditions of the test and no definite trend could be established.

LIME - FLY ASH TESTS FOR MEASURING POZZOLANIC ACTIVITY

The development of strength as a result of pozzolanic action requires that lime be available for reaction with the active constituent of a pozzolanic material. Some of the earliest methods of evaluating pozzolanic activity included determination of the effect of the reaction taking place in pozzolan-lime mixtures. Data obtained using three such procedures are shown in Table XIX.

In one method, lime - fly ash mortar was cured at temperatures of 100 and 130 F and the compressive strength determined at an age of 7 days. Details of this test as performed in this investigation are given in Procedure 3 of Appendix I. Except for fly ash samples 1, 2, and 3, higher strengths were obtained by curing the specimens at 130 F than at 100 F. The data show that the higher curing temperature is generally of marked benefit to the less active fly ashes. A comparison between the pozzolanic strength index and the compressive strength of lime - fly ash mortar cured at 100 F failed to show any correlation. A reasonable relation between the index and the strength of the lime-fly ash mortar cured at 130 F is shown in Fig. 19. However, the six fly ashes identified by number in Fig. 19 do not follow the general trend. Alexander (9) has indicated that lime mortar tests of pozzolanic materials conducted for a fixed period at constant temperature may not provide a proper measure of the reactivity of such materials. It is possible that fly ashes Nos. 4, 7, 8, 12, and 17 would have shown greater reactivity with lime if the test had been made under other conditions of storage such as higher temperature or longer curing.

TABLE XIX.—RESULTS OF LIME-FLY ASH TESTS FOR POZZOLANIC ACTIVITY.

Fly Ash Identification	Compressive Strength at 7 days of 1:2:9 Lime-Fly Ash Mortar, psi		Time of setting of 1:4 Lime-Fly Ash Mixture, hr		Lime Absorption at 28 days Less that at 7 days, CaO per 100 g of Fly Ash, g
	Cured at 100 F	Cured at 130 F	Initial	Final	
No. 1	670	580	140	313	2.25
No. 2	785	670	141	195	1.39
No. 3	925	920	52	120	1.98
No. 4	175	445	234	258	0.90
No. 5	545	860	167	192	2.17
No. 6	445	935	235	322	3.11
No. 7	325	485	177	266	0.60
No. 8	385	445	90	168	−0.23
No. 9	175	520	120	195	0.20
No. 10	490	665	216	340	3.97
No. 11	115	510	119	293	−0.03
No. 12	195	445	118	315	2.31
No. 13	95	320	340	384	−0.17
No. 14	160	490	141	374	0.32
No. 15	115	485	142	194	1.49
No. 16	235	660	119	143	1.96
No. 17	105	410	72	651	−0.04
No. 18	160	710	166	450	0.05
No. 19	65	535	507	555	0.00
No. 20	65	360	187	404	...
No. 21	80	280	260	527	1.68
No. 22	105	415	165	395	0.86
No. 23	135	425	315	482	1.75
No. 24	100	440	170	315	−0.01
No. 25	210	440	139	420	3.47
No. 26	125	620	339	651	0.09
No. 27	277	455	355	479	6.06
No. 28	130	480	216	384	1.80
No. 29	105	355	118	315	−0.17
No. 31	515	650	180	215	0.01
No. 32	110	440	340	501	−0.28
No. 33	55	335	259	404	−0.16
No. 34	175	440	381	673	2.83

The second method involved the determination of the time of setting of a lime-fly ash mixture. Moran and Gilliland (8) described this method as developed by Feret and used for evaluating pozzolanic materials other than fly ash.

The details of the test are given in Procedure 4 of Appendix I. When the initial and final setting times shown in Table XIX are compared with the pozzolanic strength index, a general trend towards an inverse relationship is found, but the relation is not sufficiently definite to make the test of much value for determining the quality of fly ash.

Moran and Gilliland (8) also described a lime absorption test to measure the activity of a pozzolan. This test provides for the measurement of the amount of lime absorbed from a saturated lime solution over periods of time. The absorption of lime was measured by the decrease in the amount of acid required to neutralize an aliquot of the solution. The difference between the amounts absorbed at 7 days and 28 days was then taken as a measure of the pozzolanic activity of the material. The details of the test as performed in this investigation are given in Procedure 5 of Appendix I. Moran and Gilliland showed good correlation between the results of this test and the strength of portland cement-pozzolan mortars when the pozzolans were volcanic ash or pumice. The results obtained with this test on the fly ashes used in this investigation are shown in Table XIX. A comparison of these results with the pozzolanic strength indexes failed to show any correlation. For a number of samples, negative values were obtained. This is not surprising as it was shown in the tests of lime-fly ash slurries that the alkalis in fly ash are slowly soluble in the presence of lime. Although the use of the 7-day result as the starting point would eliminate the effect of rapidly soluble constituents, the continued solubility of constituents in the fly ash after that time affects the equilibrium conditions with respect to lime and alkalis in solution. This test is considered not applicable to fly ashes.

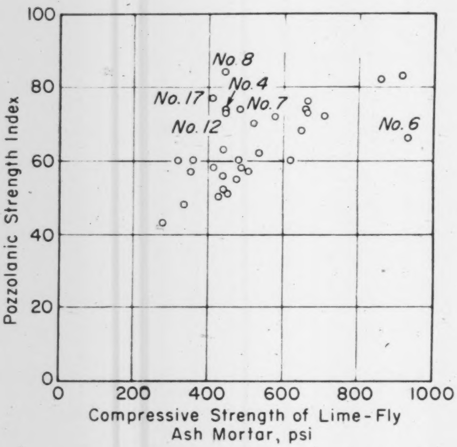

FIG. 19.—Relation Between Compressive Strength of Lime-Fly Ash Mortars and the Pozzolanic Strength Index.

Note.—Lime-fly ash mortar cured at 130 F.

TABLE XX.—RELATION BETWEEN LIMITING VALUES OF POZZOLANIC STRENGTH INDEX AND OTHER PROPERTIES OF FLY ASHES.

Number of Fly Ashes Meeting Indicated Limits	Pozzolanic Strength Index, Minimum	Carbon Content, Maximum, per cent	Loss on Ignition, Maximum, per cent	Amount Passing No. 325 Sieve, Minimum, per cent	Average Water Requirement Ratio, 35 per cent Replacement, Maximum, per cent
3......	82	3.5	4.1	93.9	98
5......	76	6.4	6.9	91.5	100
13......	70	6.9	7.5	88.9	101
15......	66	13.6	14.8	88.9	101
21......	60	13.6	14.8	75.8	109
27......	55	13.6	14.8	74.8	110
31......	50	15.6	18.0	74.8	125
33......	43	15.6	18.0	56.2	125

SIGNIFICANCE OF TEST RESULTS WITH RESPECT TO SPECIFICATION REQUIREMENTS FOR FLY ASH

The determinations made on fly ash in this study generally follow procedures used previously for testing either fly ash or other types of pozzolanic materials. The significance of each of these determinations for measuring the quality of fly ash has been assessed by comparison of the effects of fly ash on the strength of mortar. In the following discussion the test methods and specification requirements that are usually applied to fly ash are evaluated in view of the data obtained in this investigation.

The results of these tests indicate that the effect of fly ash on the strength of mortar is related to the carbon content of the fly ash, the fineness as determined by use of the No. 325 sieve or the 0.03-mm size in the hydrometer analysis, and the water requirement ratio. The use of the No. 325 sieve is preferred over the hydrometer method for determination of fineness because of the relative simplicity of the method. Limiting values for these properties for fly ashes having various degrees of pozzolanic activity are shown in Table XX. This information can be used for making a quick evaluation of a fly ash in terms of its carbon content, loss on ignition, fineness, and water requirement ratio. The data in this table may also serve as a basis for preparing a specification for fly ash for use in concrete.

Although the data in Table XX indicate that fly ashes of a selected quality as measured by the strength index will generally meet certain limits for carbon content, loss on ignition, fineness, and water requirement ratio, it should not be inferred that limits on these properties alone will always insure a particular level of pozzolanic activity. Since the relations existing between pozzolanic activity and these properties are not exact, some fly ashes may not be as active as an appraisal based on these properties would indicate. Fly ash No. 6, for example, with a loss on ignition of 4.1 per cent, 92.1 per cent passing the No. 325 sieve, and a water requirement ratio of 98 per cent, would be expected on the basis of Table XX to have a higher strength index than 66 as shown in Fig. 10. This shows that limits on these properties of fly ash do not eliminate the need for a more positive test for pozzolanic activity.

Specifications for fly ash generally include some requirement based on a test for strength of mortar prepared with cement or lime in combination with fly ash. When the mortar is prepared with lime, a minimum strength is usually specified. When cement is used, the strength of mortar containing fly ash is required to be equal to or greater than a definite percentage of the strength of a control mortar not containing fly ash. Any type I cement of acceptable quality is generally permitted in these tests. It has been shown in this investigation that the strength ratio for a particular fly ash may vary considerably when determined with different cements. Consequently, when a minimum strength ratio is specified, consideration should be given to the cements to be used in the testing procedure. The most desirable procedure would be to use cement from a single source or, if this is not feasible, to use cements which have been found to produce comparable results in mortars containing fly ash. Further research on this problem is desirable to determine whether certain properties of cement can be used for selecting such cements. There is evidence, for example, that the alkali and tricalcium silicate contents of the cement may have an important bearing on its behavior with fly ash.

The minimum strength ratio or pozzolanic strength index which should be specified depends on factors not considered in this study. The pozzolanic strength indexes of four fly ashes used in this series representing sources of fly ash which have given good results in concrete varies from 71 to 83. The feasibility of using fly ashes of poorer quality will depend on such factors as the permissible reduction which can be tolerated in the 28-day strength of the concrete and the quality of fly ashes available in a particular locality.

In the use of a lime - fly ash mortar for determining the pozzolanic activity of fly ash, control of the uniformity of the lime does not appear to be difficult. However, the results of tests of fly ash with lime did not show as close a correlation with those in which cement was used as might be desired. This may have been due to the particular procedure followed in preparing the lime - fly ash mortars, and further work with this method may furnish better results.

At present the fineness of fly ash is usually specified in terms of specific surface determined by the air permeability method. The results given in this study show that specific surface so determined has no relation to the effect of fly ash on the strength of mortar. This is in contrast to the good correlation found with the amount passing the No. 325 sieve. The use of this sieve as a means for determining the fineness of fly ash appears to be desirable.

Except for carbon, the variations in the total amounts of individual chemical constituents that are present in the samples tested in this investigation had little or no relation to the pozzolanic activity of the fly ash. This does not necessarily mean that chemical constitution is not of importance but rather that the normal variation in composition of the different fly ashes is not sufficient to produce significant changes in pozzolanic activity. It is likely that availability for reaction or the physical state of the constituents control the activity of a fly ash to a much greater extent than the total amount of constituent present. For this reason specification requirements for chemical constituents other than carbon are necessary only to limit potentially deleterious constituents or to eliminate freak materials. For fly ash, these requirements are generally limited to the percentages of silica, magnesia, sulfur trioxide, and in special cases "available" alkalis.

The present ASTM specification for fly ash[16] includes a minimum requirement for silica of 40 per cent. Of the fly ashes examined in this investigation, six would fail to meet this requirement. Three of these six have pozzolanic strength indexes greater than 70 per cent and appear satisfactory for use as admixtures in concrete. In view of this, it is believed that a slightly lower minimum silica content may be tolerated. A value of 35 per cent is suggested as being suitable.[17]

Limits for magnesia and sulfur trioxide are often included in a fly ash specification as a protection against possible deleterious effects in the concrete or mortar. This limit is usually 3 per cent in each case. The maximum magnesium oxide content found in any fly ash examined was 2.7 per cent for sample 30, which was discarded for use in the mortar tests because of its coarseness and general unsuitability for use. The remaining samples showed a maximum value of 1.4 per cent. Although a retention of the limit on magnesium oxide to prevent the inclusion of unusual materials is believed necessary, these results indicate that the magnesia content for most fly ashes is well below this limit. The sulfur trioxide content is a measure of the amount of sulfates present and varied from 0.2 to 2.8 per cent in the samples tested. A limit of 3 per cent for this material appears to be suitable.

The alkalis present in a fly ash must be considered whenever the material is used in concrete in which there is a possibility that reactive aggregate may be present. The ASTM specification for fly ash[16] includes an optional clause which limits the "available" alkalis to 1.5 per cent. As previously stated, these "available" alkalis are determined on a lime - fly ash slurry after storage at 100 F for 28 days. All of the fly ashes included in this investigation meet this requirement, the largest amount of "available" alkalis being 1.38 per cent for sample 3. However, it was also shown that in many cases the alkalis continued to dissolve after the 28-day period. It is believed that additional studies should be made to show the correlation between the results of this test and the amounts of alkalis becoming soluble in an actual mortar or concrete and whether this continued solubility is of significance to the expansion resulting from the alkali-aggregate reaction.

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[16] Tentative Specification for Fly Ash for Use as an Admixture in Portland Cement Concrete (C 350 – 54 T), 1955 Book of ASTM Standards, Part 3, p. 1281.

[17] A recent revision to the ASTM Specification for fly ash has replaced the 40 per cent minimum for silica with 70 per cent minimum for the sum of the silica, alumina and iron oxide.

CONCLUSIONS

The results of this investigation show that many factors affect the development of strength in a cement - fly ash mortar as a result of pozzolanic action. It was found that this action is influenced not only by the character of the fly ash but also by the properties of the cement. A general relation was found between pozzolanic activity and several other properties of fly ash.

The following general conclusions appear to be justified:

1. The effect on mortar strength of replacing part of the cement with fly ash varies with the cement used.

2. Compressive strength tests conducted on cement - fly ash mortars at various ages and with different cement contents indicate that ultimately fly ash has the greatest proportional benefit in lean mixes, even though tests at 28 days generally show the reverse to be true.

3. The carbon in fly ash lowers the strength of mortar because it increases the amount of water required to obtain a workable consistency, and reduces the amount of the pozzolanically active constituents of the fly ash. However, only a general relation between carbon content and mortar strength was found, since other properties of fly ash also affect the development of strength of mortar due to pozzolanic action.

4. No relation was found between the strength of cement - fly ash mortars and fineness determined by the air permeability method.

5. In general, the strength of cement - fly ash mortars varied with the fineness of the fly ash as determined with the No. 325 sieve, the finer fly ashes being associated with the higher strengths.

6. A general agreement was found between the effect of fly ash on the strength of mortar and the amount of water required to prepare the mortar. If the addition of fly ash required an increase in the amount of water needed to prepare mortar of a stated consistency, a decrease in the strength of the mortar resulted.

7. No agreement was found between the amount of any single inorganic constituent in fly ash and the efficacy of the fly ash as a pozzolanic material.

8. A method involving reaction of silica with sodium hydroxide has been used with some success for determining the quality of pozzolanic materials. This test was found to be not suitable for testing fly ash because the lime or calcium sulfate present in fly ash prevented the normal course of the reaction.

9. A lime absorption test, involving reaction of fly ash in a lime solution, gave no reliable data for predicting the pozzolanic activity of fly ash.

10. A test for the compressive strength of *lime* - fly ash mortars cured at a temperature of 130 F failed to give results agreeing satisfactorily with the strengths of *cement* - fly ash mortars.

11. Fly ash will inhibit the volume

change caused by the alkali-aggregate reaction if used in sufficient amount. Because of variations in the behavior of different fly ashes, the amount to be used with a reactive combination of materials should be determined by testing concrete composed of those materials and the particular fly ash in question.

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APPENDIX I

The following are general descriptions or details of testing procedures used in this investigation which were not included in the text but which may be of interest to those actively engaged in making similar studies.

PROCEDURE 1.—METHODS OF CHEMICAL ANALYSIS

The oxide analyses, except for the alkalis, were conducted following the general methods outlined by Hillebrand, Lundell, Bright and Hoffman in their book, Applied Inorganic Analysis—2nd Edition, John Wiley & Sons, New York, using a sodium carbonate fusion. The alkalis were determined by means of a flame photometer using the method outlined by Halstead and Chaiken in their report (**10**).

Carbon was determined by weighing the CO_2 formed by ignition of the sample at 950 C in a tube combustion furnace using oxygen. The total CO_2 absorbed was corrected for the amount resulting from carbonates by determining the CO_2 released by hydrochloric acid in a separate determination.

The sulfates (as SO_3) were determined in a portion of the sample dissolved in HCl (1:1) after 15 to 20 min. digestion at a near boiling temperature.

PROCEDURE 2.—PREPARATION AND ANALYSIS OF LIME-FLY ASH SLURRIES

The slurries were prepared as directed in the method for the determination of available alkalis.[15]

The extraction of the water soluble alkalis and the analyses of the residues were conducted as follows:

The liquid contents of the vial were transferred to a 250-ml beaker and the solid cake transferred to a mortar. The cake was ground with the addition of some water until a uniform slurry was formed and no large lumps remained. The slurry was then transferred to the same 250-ml beaker and sufficient water added to make the total volumn 100 ml.

For most samples after 28 and 91 days, the solid cakes were so hard that the vial had to be broken and the glass carefully removed before grinding. The glass was scraped as thoroughly as possible to remove the adhering fly ash and lime.

After standing for 2 hr at room temperature with frequent stirring, the slurry was filtered and the residue washed thoroughly with hot water (8 to 10 times).

The filtrate was neutralized with HCl using 1 drop of phenolphthalein as indicator. It was then transferred to a 250-ml volumetric flask and filled to the mark. This solution (or aliquot where necessary) was used to determine the amounts of sodium and potassium present by means of a flame photometer.

The flame photometer was calibrated with neutral solutions of sodium and potassium chlorides. The necessary correction for lime was made by determining the average amount present in several representative samples and using a correction curve established on that basis.

The residue from the water extraction was returned to the 250-ml beaker and 13 ml of concentrated HCl and 87 ml of H_2O added. This amount was calculated to give a final concentration of free HCl equivalent to a dilution ratio of 1:10.

The beaker and its contents were allowed to stand overnight after frequent stirring for the first several hours. The liquid was filtered into a 250-ml flask and the residue washed thoroughly with hot water. After the liquid had cooled the flask was filled to the mark and aliquots of this solution used for the determination of the acid soluble constituents.

The residue from the acid treatment was transferred to the 250-ml beaker and 100 ml of hot 1 per cent solution of sodium hydroxide added. It was then digested on a steam bath for 20 min; 5 ml of concentrated HCl was added to make the solution slightly acid and to dissolve any iron hydroxide or aluminum hydroxide which may have been released from glassy complexes by the alkali treatment.

The solution was filtered into a 250-ml volumetric flask, and the residue washed thoroughly with hot water. After the filtrate had cooled the flask was filled to the mark and aliquots of this solution used for the determination of the constituents soluble in or "solubilized" by the alkali treatment.

Photometric methods were used to determine the amounts of dissolved constituents in each solution. Silica was determined by the molybdenum blue method, and iron and aluminum oxides determined with ferron. The procedures followed were essentially the same as described by Shapiro and Brannock in *Circular 165* of the Geological Survey, Rapid Analysis of Silicate Rocks, except for necessary modifications to adjust for acid concentration.

PROCEDURE 3.—COMPRESSIVE STRENGTH OF LIME - FLY ASH SPECIMENS

The lime-fly ash mortar used for these specimens contained, by weight, one part hydrated lime, two parts fly ash and nine parts of graded Ottawa sand. Sufficient water was added to this mixture to produce a flow of 80 ± 5 per cent when determined as prescribed in the ASTM Method of Test for Compressive Strength of Hydraulic Cement Mortars.[11] The materials were mixed by hand in a 6-qt bowl. Sufficient mortar was prepared to fill two $1\frac{7}{8}$-in. diameter cylindrical glass tubes 12 in. in length which had previously been sealed at the bottom. These tubes were filled in six equal layers, each layer being tamped 25 times with a metal tamper having a plane face 1 in. in diameter.

Immediately after molding, the specimens were sealed on the top with a rosin-paraffin mixture. After storage at 73 F for 24 hr, one specimen was stored for 6 days at 100 F and one was stored at 130 F. Three hours before testing at 7 days, each specimen was cut into three equal cylinders, the glass stripped, and the cylinders capped with a sulfur capping compound. The capped specimens were kept in a moist condition at 73 F until tested in compression.

PROCEDURE 4.—TIME OF SETTING OF LIME - FLY ASH SPECIMENS

A paste consisting of one part of hydrated lime and four parts of fly ash by weight was prepared with sufficient water to produce a paste of normal consistency as determined by the Method of Test for Normal Consistency of Hydraulic Cement.[18] This paste was placed in a cylindrical container of about 3-in. diameter, and covered with a layer of saturated lime water, which in turn was covered with a film of lubricating oil to prevent carbonization.

At intermittent intervals, the paste was tested by means of the Vicat needle to determine when initial and final set occurred. Initial set was considered to have occurred when the 1 mm needle penetrated to 35 mm from the upper surface of the paste in 30 sec. When the needle showed no penetration of the surface of the paste, final set was concidered to have taken place.

PROCEDURE 5.—LIME ABSORPTION TEST

This test is intended to determine the capacity of a fly ash for reacting with lime in a lime water solution. The following procedure was used:

A 1-g sample of fly ash was placed in a test tube to which was added 100 ml of a lime water solution of known concentration as determined by titration with a standardized 0.04*N* hydrochloric acid solution. The test tube was sealed and agitated constantly by mechanical means until the date of test. At ages of 7 and 28 days, a 25 ml portion of the supernatant liquid was removed and immediately titrated with the 0.04*N* hydrochloric acid solution. After the sample for test at an age of 7 days had been taken, the solution in the test tube was replenished with 25 ml of lime water of the same concentration as originally used.

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[18] Method of Test for Normal Consistency of Hydraulic Cement (C 187 – 49), 1952 Book of ASTM Standards, Part 3, p. 152.

The amount of lime absorbed by the fly ash was determined by the change in alkalinity of the solution during each period of agitation as indicated by the amount of $0.04N$ hydrochloric acid needed for titration.

APPENDIX II

The following discussion[19] is based on a number of published references and, while there is not always complete agreement on all details, it is believed that it represents generally the concensus of thought concerning the chemical constitution of the inorganic matter in coal and the changes which occur during combustion to form fly ash.

The bulk of the inorganic material in coal is derived from detrital or adventitious matter which fills the fissures and cracks occurring in coal beds. This material consists mainly of silicates, sulfides, and carbonates. The siliceous matter is mostly shale and clay, kaolin being the most abundant mineral present. Smaller amounts of potash clays, micas, feldspars, and quartz may also be present. The shales are largely complex alumino-silicates in combination with small amounts of iron, calcium, sodium, and potassium. The sulfide minerals present are essentially in the form of pyrite (FeS_2) and marcasite (FeS_2). Calcite ($CaCO_3$) and siderite ($FeCO_3$) make up the bulk of the carbonate minerals occurring in coal beds. It has been estimated that more than 90 per cent of the mineral matter in coal consists of kaolin, calcite, pyrite, and clay.

The grinding of pulverized fuel largely separates the coal from the associated minerals and even the minerals from each other into discrete particles. As the particles are burned in an air suspension, collisions are infrequent and the minerals are not again mixed. Thus, their residues appear separately in the fly ash.

When bituminous coals are used as the pulverized fuel, the small particles of coal in suspension are heated rapidly. The particles soften and swell into multicellular bubbles of coke, known as "cenospheres." Some particles of bituminous coal may increase in volume by as much as 40 times shortly after entering the furnace, the average being about 11 times the original volume. The density of coal (1.3 to 1.7) is thereby decreased to approximately 0.12 to 0.15 by cenosphere formation. Much of the inorganic matter in coal is finely divided. The swelling of the coal further separates and subdivides the ash into minute particles. As the cenospheres burn, numerous bits of mineral ash are released.

The mineral matter undergoes considerable decomposition and chemical change during combustion, and the chemical nature of the ash is quite different from that of the original mineral matter in coal

Each of the three main minerals (kaolin, calcite, and pyrite) break down to their respective oxides at the temperature of the fuel bed (980 to 1650 C), but the resulting oxides usually have melting points above the combustion temperature. However, eutectic relationships are formed which result in fusion.

In the flame, the shale and clay particles lose combined water and those particles which leave the flame prematurely do not complete the processes of fusion or that of moisture evolution. These particles are white and rounded, the whiteness being due to cavities formerly occupied by steam. The particles which continue in the flame tend to complete both processes particularly in relatively hot combustion chambers and they form complex silicates that are colorless, solid spheres resembling glass. Various tints of color may be due to iron components in the shale.

The pyrite (FeS_2) is ignited to the oxide state with the accompanying evolution of sulfur dioxide (SO_2) and sulfur trioxide (SO_3). If the temperature is below 1000 C, ferric oxide (Fe_2O_3) forms. However, the most likely situation is that ferroso-ferric oxide or magnetite (Fe_3O_4) is formed, since temperatures usually exceed 1000 C. This material is in the form of black rounded or spherical particles and are often mistaken for particles of carbon. These particles are highly magnetic.

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[19] This discussion was prepared by B. Chaiken, Chemist of the Physical Research Branch, Bureau of Public Roads.

The calcite, of course, breaks down to calcium oxide and carbon dioxide during ignition. Usually the bulk of free lime so produced combines with the sulfur dioxide and trioxide evolved from the pyrite to form calcium sulfate. Generally, the sulfur oxides are fixed as sulfates by any free particles of basic oxides. Free lime not so converted to sulfates will remain as calcium oxide or be converted to calcium hydroxide during storage.

The unburned coal particles which rise to the stack will be found in the fly ash as large, gritty, porous, irregularly shaped particles of cindery coke.

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DISCUSSION

Mr. L. John Minnick.[1]—The paper refers to a previous publication[2] which covered some work which was done by Sub-committee III-h of ASTM Committee C-9 on Concrete and Concrete Aggregates[3] and by members of utilities who supplied most of the results. It is gratifying to note that the conclusions reached by the present authors agree substantially with those presented in the other studies.

I would like to present three points in connection with the paper. First, with respect to the water requirement studies, it is felt that this is a matter of considerable importance and, as the authors point out, probably has as much or more bearing on the performance of fly ash in concrete than does the presence of residual carbon. Our investigations revealed that removing larger particles (which may or may not be rich in carbon), such as those which are retained on a No. 200 sieve, apparently removes from the fly ash that material which tends to produce the higher water content. Thus, tests run with the finer fraction material show that this fly ash has a lower water requirement. A few results given in the accompanying Table XXI show the improvement obtained. The graph shown in the accompanying Fig. 20 also indicates that there is a definite relationship between compressive strength and the water requirement. The plotted points include different fly ashes as well as various fractions of the individual samples. It is thus indicated that it may be possible to take fly ashes which are considered to be unsatisfactory for portland-cement concrete and convert them to materials that are acceptable.

The second point relates to the chemical tests mentioned by Mr. Brink. The accompanying Tables XXII and XXIII show the results obtained from tests that have been run with additions of hydrated lime to portland-cement mortars and concrete. It has been found that with certain cements it is possible to introduce small amounts of hydrated lime and thereby improve the strength of the resulting concrete at early ages. The hydrated lime probably supplements the action developed by the release of the hydrated lime by the portland cement—some cements yielding more lime than others. The presence of sodium hydroxide, calcium sulfate, and other soluble materials also has a strong bearing on the performance of fly ash in concrete.

The final point is in regard to the enthusiasm that these authors have for the use of fly ash. While it is agreed that fly ash is of considerable value in concrete, we feel that a 33⅓ or 50 per cent replacement of portland cement, especially in applications involving high-

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[1] Vice-President in charge of Research, G & W. H. Corson, Inc., Plymouth Meeting, Pa.

[2] L. John Minnick, "Investigations Relating to the Use of Fly Ash as a Pozzolanic Material and as an Admixture in Portland Cement Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 54, p. 1129 (1954).

[3] On Methods of Testing and Specifications for Admixtures.

TABLE XXI.—RELATIONSHIP BETWEEN SIEVED FRACTIONS OF FLY ASH AND PHYSICAL PROPERTIES OF CEMENT-FLY ASH MORTARS.[a]

Fly Ash Sample	Sample Description	Weight for Each Fraction, per cent	Ignition Loss, per cent	Water[b] Requirement	Compressive Strength, per cent of control	
					7 days	28 days
No. 1.....	As received	100.0	3.14	112	84.5	94.9
	Passing No. 200 Sieve	75.5	3.37	104	108.4	120.1
	Passing No. 100 Sieve	89.4	3.74	...	...	...
	Retained on No. 200 Sieve	24.5	3.62	...	...	...
No. 2.....	As received	100.0	23.60	123	76.0	83.0
	Passing No. 200 Sieve	61.3	11.40	104	118.0	122.0
	Passing No. 100 Sieve	86.8	19.40	110	96.0	104.0
	Retained on No. 200 Sieve	38.7	43.80	170	28.3	30.4
No. 3.....	As received	100.0	8.50	98	129.0	149.0
	Passing No. 200 Sieve	94.1	7.49	94	154.0	169.0
	Retained on No. 200 Sieve	5.9	24.97	...	...	...
No. 4.....	As received	100.0	8.42	108	80.6	100.0
	Passing No. 200 Sieve	81.5	5.26	106	90.4	112.0
	Retained on No. 200 Sieve	18.5	22.40	...	...	...

[a] Designed and tested in accordance with requirements of ASTM Method C 311.[4]

[b] Reported as per cent of control specimens which do not contain fly ash.

TABLE XXII.—EFFECT OF ADDITION OF HYDRATED LIME ON COMPRESSIVE STRENGTH OF CEMENT-FLY ASH MORTARS.[a]

Portland Cement Sample[b]	Fly Ash Sample[c]	Lime Addition by weight of Fly Ash,[d] per cent	Compressive Strength, psi[e]	
			7 days	28 days
No. 1..............	...	...	5495 ± 125	6789 ± 37
No. 1..............	No. 1	...	4488 ± 222	6751 ± 280
		2.0	5355 ± 50	6864 ± 286
No. 1..............	No. 2	...	4053 ± 76	6493 ± 158
		2.0	4931 ± 140	7526 ± 214
No. 1..............	No. 3	...	4963 ± 207	7148 ± 405 (2 tests)
		2.0	5418 ± 256	7823 ± 159
No. 2..............	...	...	5104 ± 65	7112 ± 3
No. 2..............	No. 4	...	5210 ± 162	6976 ± 193
		2.0	5221 ± 40	6909 ± 249

[a] Mix proportions: 1 part cement to 2.75 parts by weight of commercial mortar sand (fineness modulus = 2.79). Fly ash—25 per cent replacement of cement by absolute volume.

[b] Pennsylvania Highway Dept. type C portland cement. Supplied from two different mills.

[c] Fly ash: Sample No. 1: Low carbon, medium fineness
Sample No. 2: High carbon, high fineness
Sample No. 3: Low carbon, high fineness
Sample No. 4: Medium carbon, medium fineness

[d] High-calcium hydrated lime passing ASTM Specification C 207.[5]

[e] Average of 3 tests.

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[4] Tentative Methods of Sampling and Testing Fly Ash for Use as an Admixture in Portland Cement Concrete (C 311 - 54 T), 1955 Book of ASTM Standards, Vol. 3, p. 1332.

[5] Specification for Hydrated Lime for Masonry Purposes (C 207 - 49), 1955 Book of ASTM Standards, Vol. 3, p. 215.

TABLE XXIII.—EFFECT OF ADDITION OF HYDRATED LIME ON COMPRESSIVE STRENGTH OF FLY ASH CONCRETE.[a]

Sample	Fly Ash Addition, lb per cu yd	Lime Addition, lb per cu yd	Compressive Strength, psi[b]	
			7 days	28 days
Control	None	None	1760 ± 20	3235 ± 105
No. 201	200	None	1387 ± 200	3130 ± 365
No. 202	200	25	1775 ± 71	3336 ± 250
No. 203	200	50	1997 ± 71	3656 ± 109

[a] Design: Control mix

Portland cement	440 lb per cu yd
Fine aggregate	1575 lb per cu yd
Coarse aggregate	1830 lb per cu yd
Fly ash mix	
Portland cement	350 lb per cu yd
Fly ash	200 lb per cu yd
Fine aggregate	1475 lb per cu yd
Coarse aggregate	1830 lb per cu yd

Slump, approximately 6 in.
Lime added to fly ash mix as straight addition.
Portland cement—Pennsylvania Dept of Highways type C.

[b] Average of 3 tests.

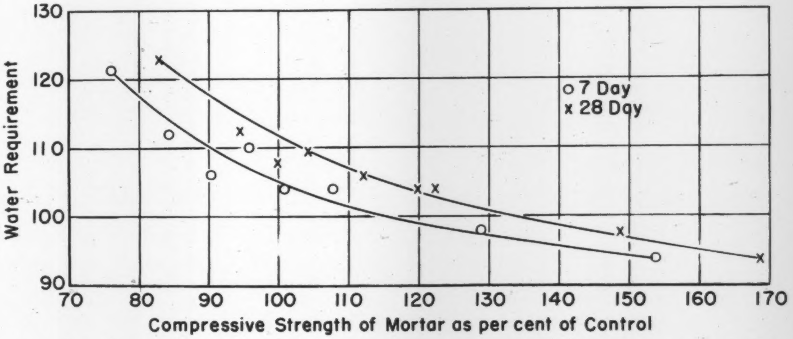

FIG. 20.—Relationship Between Water Content of Cement-Fly Ash Mortar and Compressive Strength.

way concrete, might easily be considered to be excessive. Retaining a reasonably high cement factor seems more advisory, although the fly ash content should also be ample. The proper design of concrete usually requires fly ash over and beyond that which is used merely to replace a smaller amount of portland cement.

MR. R. C. MIELENZ.[6]—I should like to ask three general questions: First, is there any relationship between the potash content and any properties of the mortar or concrete?

MR. W. J. HALSTEAD (*author*).—We compared the potassium oxide content, as well as the other chemical constituents, with the strength index but found no significant relation.

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[6] Formerly, Head of Petrographic Laboratory, Bureau of Reclamation, Denver, Colo. Currently, Director of Research, The Master Builders Co., Cleveland, Ohio.

MR. MIELENZ.—Was there any relation with the volume change (shrinkage) of mortar?

MR. BRINK.—We did not make mortar tests of that type.

MR. MIELENZ.—Was there any correlation between the performance of the fly ash and the content of silica, alumina, and ferric oxide? These three components are combined in the latest draft of the specifications on natural pozzolan.

MR. HALSTEAD.—We did study the relation of the strength index to various combinations of silica and alumina but we did not include the iron oxide. We were unable to show any definite correlation.

MR. MIELENZ.—Was any attempt made to correlate the activity of the cement-fly ash combinations with the C_3S content of the portland cement?

MR. BRINK.—The strength tests to which I referred were made up to an age of one year. Actually we made strength tests at 3, 7, 14, 28, 91, and 365 days and we studied the relations between the strengths at these ages and various properties of the cements. At an age of one year, there seemed to be some relation between the C_3S content of the cement and the strength developed. We have gone into detail on that subject in our paper.

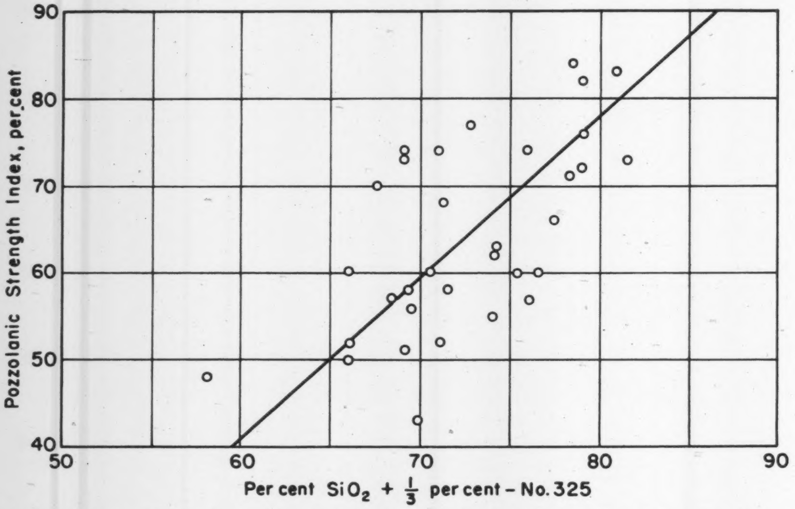

FIG. 21—Relation of Pozzolanic Strength Index to Silica Content Plus Fineness of Fly Ash.

MR. BRYANT MATHER.[7]—In Fig. 11 of Messrs. Brink and Halstead's paper four kinds of data on fineness of fly ash are plotted against pozzolanic strength index. The authors state that only the amount of material passing the No. 325 sieve showed a reasonably definite relation to pozzolanic strength index. In Fig. 15 of the same paper, silica content is plotted against pozzolanic strength index and the authors state that no indication of a definite relation was found in spite of the fact that it might be expected from the definition of a pozzolan that this "constituent" would have some effect on the strength of mortar.

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[7] Engineer, Concrete Research, Concrete Division, Waterways Experiment Station, Corps of Engineers, U. S. Army, Jackson, Miss.

It is my belief that a large number of factors affect the actual degree to which a given quantity of pozzolan will contribute compressive strength in a given mortar at 28 days but that, other things being equal, the type and quantity of silica and the state of subdivision of the material will be most important. In tests of any single class of pozzolans, such as fly ash, it may be assumed that at least most of the silica is of the same type from sample to sample. It would therefore be expected that pozzolanic strength should correlate with some combined effect of silica content and fineness. Figure 21 shows the relation between average pozzolanic strength index (taken from Fig. 10 of Messrs. Brink and Halstead's paper) and the combined effect of silica content (taken from Table I) and per cent passing No. 325 sieve (taken from Table II of the same paper, respectively). Since silica content values are in the range 33 to 52 per cent and values for per cent passing No. 325 in the range 57 to 99 per cent and since I expect that silica content might be expected to have a greater effect on strength than fineness, the parameter selected to represent the combined effect is: per cent $SiO_2 + \frac{1}{3}$ per cent finer than No. 325. It appears from inspection that the relation shown in Fig. 21 is somewhat better than that shown in either Fig. 11 or Fig. 15 of Messrs. Brink and Halstead's paper, for the two factors taken separately.

MRS. KATHARINE MATHER.[8]—The discussion in Appendix II of Messrs. Brink and Halstead's paper is particularly interesting. Our studies of four samples of fly ash, partially reported in Tables XVI to XVIII of the paper by Minnick,[2] revealed the presence of the following crystalline constituents in all four: quartz, mullite, magnetite, and hematite. Mullite is apparently to be expected in many systems containing SiO_2, Al_2O_3, and a third constituent. Mullite as a constituent of fly ash is mentioned by Eitel[9]; it is the usual high-temperature product from kaolin, which, as the authors note, is the major clay mineral associated with American bituminous coals. Brownell[10] indicates that K_2SO_4 would be the sulfate formed until the K^{++} ion was used up, then $CaSO_4$ would be formed until either the calcium ion or the sulfate ion were used up, and finally Na_2SO_4 would form if there were remaining sulfate ion.

MESSRS. R. H. BRINK AND W. J. HALSTEAD (*author's closure*).—We find the written discussions submitted by Messrs. Minnick and Mather and Mrs. Mather very interesting and are gratified that our data has prompted such comment and additional study. We feel that much of the value of our paper is to provide the type of basic information that will be useful in further research on fly ash.

Mr. Minnick seems to have interpreted our replacement of 35 and 50 per cent of the cement with fly ash in mortar test specimens as an enthusiasm for the use of such large replacements in concrete for highways. The inclusion in our work of replacements as high as 50 per cent does not imply that we are recommending the use of fly ash to such an extent in concrete, since the proper design of concrete containing fly ash does not lie within the scope of this paper. However, we do believe that such large replacements are useful for research purposes in order to accentuate the full effect of each fly ash on the strength or other properties of mortar.

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[8] Chief, Petrography Section, Concrete Division, Waterways Experiment Station, Corps of Engineers, U. S. Army, Jackson, Miss.

[9] W. Eitel, "The Physical Chemistry of the Silicates," University of Chicago Press, p. 1133 (1955).

[10] W. E. Brownell, "Fundamental Factors Influencing Efflorescence of Clay Products," *Journal*, Am. Ceramic Soc., Vol. 32, pp. 375–389 (1949).

We agree completely with Mr. Mather in the belief that a large number of factors affect the degree to which a pozzolanic material will contribute to compressive strength. We also agree that, "other things being equal," the type and quantity of silica and the state of subdivision of the material will be important. However, we should keep in mind that for this investigation the fly ashes tested varied in other respects besides silica and fineness and therefore "other things" were not equal. Any apparent correlation or lack of correlation results from a combination of a large number of factors.

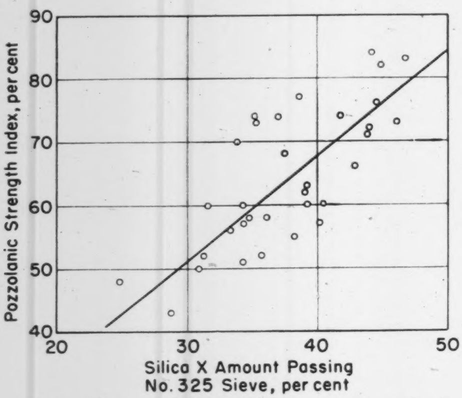

FIG. 22.—Relation Between Pozzolanic Strength Index and the Amount of Silica Passing the No. 325 Sieve.

Although not reported in the paper, we made studies of a number of combinations of silica, alumina, and fineness. These took into account molecular ratios, possible combinations in the end product, and the calculated amounts of various constituents passing the No. 325 sieve. None of these gave correlations that were considered to be significantly better than the correlation with fineness alone.

It did not occur to us to consider the particular combination of silica and fineness suggested by Mr. Mather (SiO_2 + $\frac{1}{3}$ the amount passing a No. 325 sieve) but we did study the correlation of strength with the product of the silica content and fineness. Unlike Mr. Mather's parameter, such a product has the virtue of quantitative significance in that it represents the amount of silica passing the No. 325 sieve if the assumption is made that the chemical composition of the fraction finer than this sieve is the same as that for the fraction retained. The plot of this product *versus* the pozzolanic strength index, Fig. 22, is quite similar to that shown by Mr. Mather. The coefficients of correlation obtained between several of the parameters and strength are as follows:

Parameter	Coefficient of Correlation, r	Probable Error of Coefficient
Amount passing No. 325 sieve, per cent	0.86	±0.0304
SiO_2, per cent	0.21	±0.1123
SiO_2 + ⅓ amount passing No. 325, per cent	0.64	±0.0693
SiO_2 + amount passing No. 325, per cent	0.85	±0.0321
SiO_2 × amount passing No. 325, per cent	0.75	±0.0507

Except when silica is considered alone, the values for coefficient of correlation would indicate that each of the remaining parameters have a highly significant correlation with strength. The possible combinations of silica and fineness which will yield coefficients of high significance are apparently unlimited and yet none were found to have better correlation with the strength index than the amount passing the No. 325 sieve alone.

In connection with Appendix II of our paper, Mrs. Katharine Mather calls our attention to studies that show quartz, mullite, magnetite, and hematite as four crystalline constituents generally found in fly ash. Appendix II was presented as a general review of the literature for background information. The information available to us at the time was not precise concerning the constitution of the silicates formed; thus we merely referred to these as "complex silicates" and made no attempt to further define their mineralogical character.

After the matter of the presence of mullite was called to our attention, X-ray diffraction patterns were made for fly ashes Nos. 1, 3, 6, 14, 29, and 31. The patterns for mullite along with those for quartz and magnetite were very definite for each of these samples. There was also some indication of the presence of hematite, but most of the iron was indicated to be magnetite. Therefore, this later work confirms the findings of Mrs. Mather and others.

The work by Brownell[10] quoted by Mrs. Mather might indicate that we should expect a large proportion of the sulfate to be present as potassium sulfate rather than calcium sulfate as shown in our Table X. However, the experimental data from which the results in this table were calculated positively indicate that no large amounts of alkali sulfates were present. The amounts of water-soluble potassium and sodium compounds are very small in comparison with the total amounts of these constituents present (Table I). It is recognized that the water-soluble sodium and potassium may have been actually present as sulfate rather than hydroxide as calculated, but the amounts of these alkalies are such that it makes no significant difference whether they are calculated as sulfate or hydroxide. The X-ray diffraction patterns previously mentioned support the evidence obtained from the chemical analyses as the presence of the calcium sulfate lines were easily discernible for all the fly ashes examined except for samples 14 and 29. It will be noted that the calculated amounts of $CaSO_4 \cdot 2H_2O$ in these two samples were about 1 per cent in each case, and therefore the failure to detect these small amounts is not surprising.

It is also of interest to note that although Brownell[11] indicated the order of combination with sulfate to be potassium, sodium, and calcium respectively when all components were readily available, his work with complex silicates such as shales showed that the alkalies so combined did not react extensively with sulfate during firing even at relatively high temperatures. Therefore, while it is likely that some of the alkali minerals of the coal ash are broken up during the burning, it is shown that the conditions are such that stable combinations with sulfate do not occur.

MR. C. E. LOVEWELL (*by letter*).[11]—Three or more years of testing 34 fly ashes as to chemical and physical characteristics serve as adequate background information for evaluating fly ash. The authors are to be commended for a thorough job well done. Of particular value is the conclusion that "the strength developed in portland-cement - fly ash mortars is related to the carbon content of the fly ash, the fineness of fly ash as measured by the amount passing the No. 325 sieve, and the water required for mortars containing fly ash as compared with the water required for similar mortars without fly ash." We think these significant statements should help guide ASTM committees in future revisions of ASTM Specification C 350 - 54 T.[12] In our opinion, these simple tests have greater significance than some of the chemical limitations currently contained in that specification. As a matter of fact, the proper ASTM committee now has under consideration a raise in allowable SO_3 content, based on tests made with fly ashes having a range in SO_3 content from 0.94 to 17 per cent. The present limit is 3 per cent.

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[11] Sales Manager and Service Engineer, Chicago Fly Ash Co., Chicago, Ill.

[12] Tentative Specification for Fly Ash for Use as an Admixture in Portland Cement Concrete (C 350–54T), 1956 Book of ASTM Standards, Part 3, p. 1281.

There also is much discussion in committee concerning elimination altogether of certain chemical limitations as having little significance in favor of the tests on loss on ignition, percentage passing the No. 325 sieve and comparative water demand. This paper has a wealth of information to support such a position.

THE EFFECTS OF REVIBRATION ON PROPERTIES OF PORTLAND CEMENT CONCRETE*

By D. H. Sawyer and S. F. Lee[1]

Synopsis

The study was designed to investigate the effects of delayed revibration on the physical properties of concrete. Samples of concrete, with and without air entrainment, were revibrated after delays ranging from 45 min to 6 hr; and properties of the concrete were compared afterwards. Comparisons were also made with the strengths obtained from standard methods of making and curing laboratory specimens without revibration.

The test results indicate that strengths are substantially increased by revibration after delays up to 5 hr for the air-entrained and 6 hr for the non-air-entrained concretes. The durability of the non-air-entrained concrete was critically decreased by revibration, while that of the air-entrained was not significantly affected. Results of permeability tests varied over such a wide range that the data from them could not be evaluated.

At least since the first patent on portland cement, in 1824, the concept that the set of "green" concrete should not be disturbed has prevailed among engineers. No information is known to be available in technical publications that indicates investigation of this traditional opinion; and it is still largely held today that any disturbing of the initial hardening process of concrete is an unsound practice. The opinion is reflected in such standard requirements as the limited period of time permissible between the mixing and placing of concrete, and by the limiting of time between pourings of successive batches.

Yet, for years, some engineers have, upon occasion, found it necessary to disturb the hardening process, often with no apparent detrimental effects upon the properties of the finished job. This has happened when the delivery of concrete to a partially complete pouring has been somehow delayed, thus presenting the problem of avoiding cold joints. In many such instances, the problem has been overcome by having workmen tamp and agitate the surface of the concrete, often for several hours, until the fresh concrete has arrived. The practice has often been quite successful, in direct contradiction to the traditional opinion concerning the hardening process.

Also, the idea of intentionally disturbing the early hydration process of concrete in actual practice is not altogether new. During World War II the shipbuilding industry used a revibration technique to great advantage in obtaining

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

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watertight concrete in the building of concrete tankers and drydocks. The technique was adopted in order to release the water which is generally trapped beneath horizontal reinforcing bars; and not only was this achieved by the disturbance of the hydration process, but also many of the properties of the concrete appeared to be actually improved by the revibration.

Finally, the Bureau of Reclamation, in the Sixth Edition of its Concrete Manual, states that "Revibration has been found to be beneficial rather than detrimental, provided that the concrete is again brought to a plastic condition." The manual further states that "Revibration may be accomplished by immersion-type vibrators, by form vibration, or by transmittal of vibration through the reinforcement system."

In the light of the apparent contradiction between general traditional opinion and the above-mentioned exceptions to that opinion, and considering the potentialities involved in the possible evolution of a revibration technique, the Research Division of the Kentucky Department of Highways undertook a series of controlled experiments with the intent of determining the effects of revibration on the properties of portland-cement concrete. The intent of the study was not so much to consider variation in mix designs, water-cement ratios, and the like, but to discover the extent to which the hydration process in a given mix design could be interrupted to advantage, or at least without undue concern.

The observations to be reported in this paper were made on a single mix design, both with and without air entrainment. Samples of a 6-bag non-air-entrained concrete mix were given normal vibration at the initial casting and then revibrated after progressively greater delays—in intervals in multiples of 45 min—up to and including 6 hr after the initial vibration, after which delay it was found no longer feasible to revibrate. Samples of a 6-bag air-entrained mix were treated in the same manner, except that it was found no longer feasible to revibrate them after delays greater than $5\frac{1}{4}$ hr, primarily because of the decrease in water content consequent from air entrainment.

The two control groups of specimens were used in order to permit adequate comparison with traditional techniques: samples compacted by hand rodding, and samples undergoing only an initial vibrating process. All specimens were given parallel tests; the data were recorded and then tabulated.

Later investigations were begun in order to develop data on a 4.5-bag mix and on specimens with a retardant used to delay the setting time, thus permitting revibration after delays up to 16 hr after casting. Most of the tests in this latter series have been made; but, unfortunately, they will not have been carried to completion in time for inclusion in this paper.

MATERIALS

The conditions of tests and properties of the individual materials used were assumed to be constant throughout the entire investigation. The cement was regular portland (type I), purchased in bags received in local shipments at various intervals during the investigation. Air entrainment in certain mixes was obtained by the admixture of commercial NVX.

Laboratory and field performance records for the limestone coarse aggregate (local source) used in all mixes have shown that the durability characteristics of this material are excellent. The fine aggregate used—a material dredged from the Ohio River, near Louisville—was also considered of satisfactory quality. Physical properties of these aggregates and the required gradations were as noted in Table I.

TABLE I.—PHYSICAL PROPERTIES OF AGGREGATES.

LIMESTONE COARSE AGGREGATE	
Bulk (oven dry) sp gr	2.72
Bulk (saturated surface dry) sp gr	2.74
Absorption, per cent	0.50
Los Angeles abrasion, per cent	26.0
Soundness, Na_2SO_4, per cent	3.0

Sieve	Per Cent Passing
1½-in.	100
1-in.	85–95
½-in.	25–60
⅜-in.	5–25
No. 4	0–5

FINE AGGREGATE	
Bulk (oven dry) sp gr	2.64
Bulk (saturated surface dry) sp gr	2.66
Absorption, per cent	0.60
Fineness modulus	2.60

Sieve	Per Cent Passing
⅜-in.	100
No. 4	85–100
No. 16	40–80
No. 50	2–30
No. 100	0–5

TESTING PROCEDURES

Methods of proportioning materials, mixing, curing and testing of the concrete were sufficiently related to current accepted practices that they need not be discussed in detail.

All concrete was mixed in a small pan mixer of 2.0 cu ft capacity and cast into 6 by 12-in. cylinders and 3 by 5 by 20-in. beams. Specimens were cured under standard moist room conditions until the specified test age. Mix proportions were obtained by the solid volume method, using appropriate b/b_0 factors. This method suggests certain empirical relationships between the quantity b/b_0 and the fineness modulus of the sand and size of the coarse aggregate used.

Specimens for compressive and flexural strength testing were tested at ages of 7, 28, and 90 days. Corresponding durability specimens were permitted to cure 14 days in a moist room prior to beginning the freezing-and-thawing test. Three specimens were made for each set of test conditions.

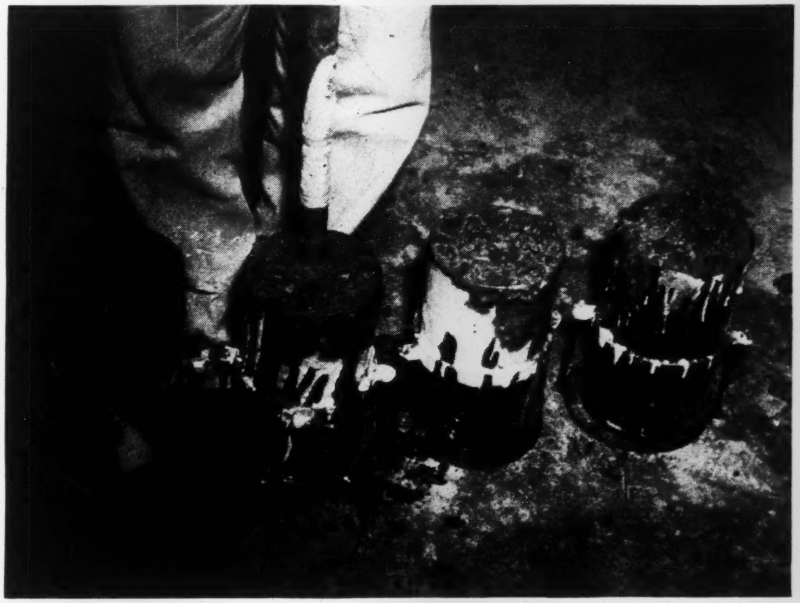

FIG. 1.—Concrete Undergoing Revibration in Cylinder Molds After a Delay of 5¼ hr. Specimens were Revibrated by Inserting the Vibrator for 4 to 40 sec at each of Three Locations Depending upon Interval of Delay.

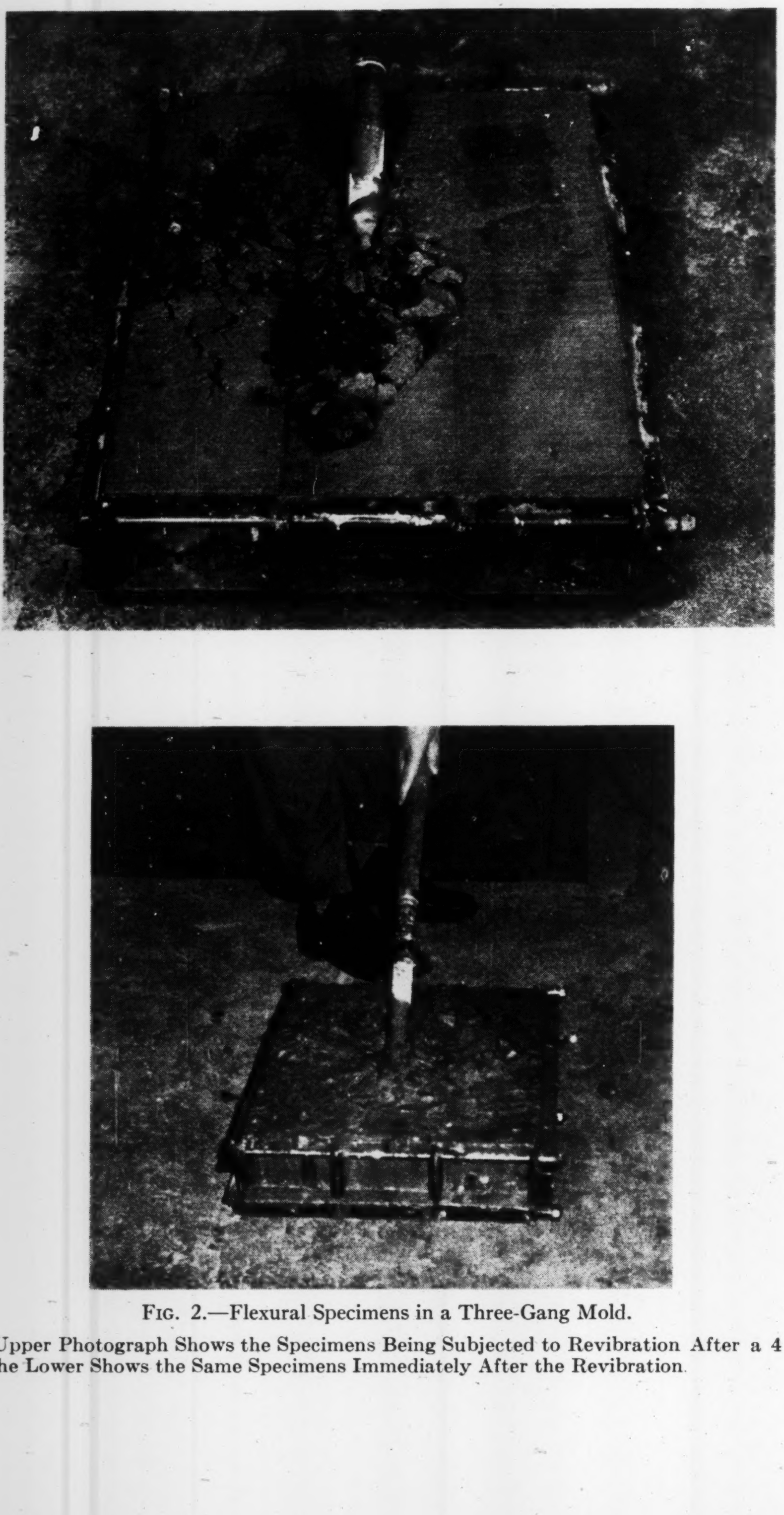

FIG. 2.—Flexural Specimens in a Three-Gang Mold.

The Upper Photograph Shows the Specimens Being Subjected to Revibration After a 4 hr Interval; the Lower Shows the Same Specimens Immediately After the Revibration.

Revibration Technique:

All prepared test specimens were kept in the molds at room temperature under damp burlap for 24 hr. The vibrator used had a $1\frac{1}{4}$-in. by 12-in. head, which vibrated at a frequency of 10,000 cpm. The procedure involved in the actual vibration was held constant for the most part; however, after the greater intervals of delay it was necessary to increase the vibration time progressively in order to obtain the desired consistency. Specimens were vibrated for periods of 12 to 120 sec each, by three equally spaced insertions of a vibrator into the molds. Compressive and flexural strength specimens being subjected to the revibration are shown in Figs. 1 and 2.

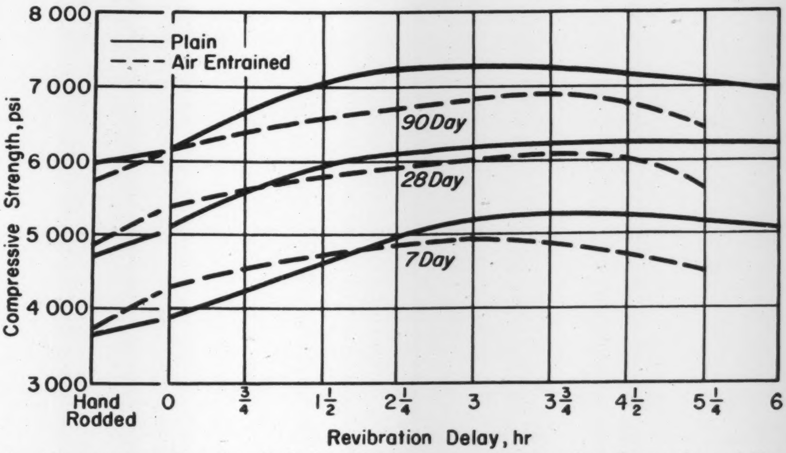

FIG. 3.—Trend Lines Showing Relationship Between Compressive Strength and Revibration Delay for Concrete With and Without Air Entrainment.

Freezing and Thawing:

The durability specimens were tested by the ASTM rapid freeze in air and thaw in water method (C 291 – 52 T).[2] This test was not conducted in strict accordance with the normally prescribed method, however, as the time required for completing each cycle was somewhat in excess of that specified. Although the equipment used was adequate, the relatively large specimens (3 by 5 by 20 in.) prohibited short cycles. The periods required for freezing the specimens to 0 F and thawing to 40 F were of approximately $3\frac{3}{4}$ hr and $1\frac{3}{4}$ hr, respectively. The capacity of the equipment, which is completely automatic, was 48 beams. Periodic determinations of the dynamic modulus of elasticity were made to measure the rate of deterioration of each specimen.

EFFECTS OF REVIBRATION

The method used in vibrating and revibrating the specimens was the same in all cases. During initial casting, and after a specified time had elapsed, the vibrator was inserted in the specimen at three different locations. The length of time for each of the three insertions varied progressively from about 4 sec to a total of 40 sec of vibration as the amount of delay was increased. During the initial casting of the specimens, the molds were filled approximately $\frac{3}{4}$ full for the first insertion, and additional concrete was added to the mold for the second and third. Upon completion of casting, an excess of concrete was placed on the individual specimens to allow for consolidation at revibration. In some instances it was necessary to add additional material in order that the specimens have the desired height. This was generally limited to the longer revibration periods and could have been eliminated entirely by use of a short extension or band clamp around the tops of the molds. In actual practice, if revibration were being used in single thickness or one layer construction, it would be necessary to provide forms of slightly greater depth than actually required. Under other applications, however, an excess of concrete would probably not be needed. For example, if this technique were employed as a means of eliminating cold joints in bridge abutments, structures, etc., it would not be necessary to account for the volume change if the final layer of concrete to be poured were not revibrated.

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[2] Tentative Method of Test for Resistance of Concrete Specimens to Rapid Freezing in Air and Thawing in Water (C 291 – 52 T), 1955 Book of ASTM Standards, Part 3, p. 1343.

Compressive Strength:

The influence of revibration on the compressive strength is probably as significant to an understanding of the effects of this technique as the effects on any of the other variables considered. Results of the compressive strength data for the different mixes are shown in Fig. 3. The strengths are expressed in terms of the revibration delay—the time elapsed between casting and revibration. The data also include results from the specimens which were hand rodded and from those which were given only initial vibration.

In the case of the non-air-entrained concrete it was possible for the mix to regain a satisfactory degree of plasticity and workability after a 6-hr delay. With the air-entrained concrete, however, it was impossible to disrupt the hydration process beyond $5\frac{1}{4}$ hr. The maximum delay feasible appeared to be governed by the water-cement ratio, temperature of the concrete, and the total volume made.

The greatest increase in compressive strength of revibrated concrete above that of the non-revibrated concrete was for the 7-day specimens without air entrainment. This increase was approximately 36 per cent and occurred in specimens revibrated after a delay of about 4 hr. The corresponding increases for the 28- and 90-day specimens were 24 per cent and 15 per cent, respectively. The increase in strength of the air-entrained specimens was approximately half that of the non-air-entrained at each age test. The maximum increase in strength for specimens containing air entrainment occurred when revibration had been given about 3 hr after casting. The increases in strength for the 7, 28, and 90-day tests were 16, 13, and 9.5 per cent, respectively. Thus, the influence of revibration on these specimens resulted not only in an increase in strength through consolidation but also served to accelerate the hardening process.

Specimens involved in the 4.5-bag mix design and those made with the retardant have not attained sufficient age to warrant inclusion or discussion at this time. Nevertheless, several of these specimens have been tested and they offer additional support for the data obtained from the 6-bag mix. In this instance the effect of revibration appears much less significant, but emphasizes, in a general way at least, the importance and controlling influences of the richer mortars. These influences seem to lie within the framework of the richer mortars which are further enhanced by revibration during the early dissolution

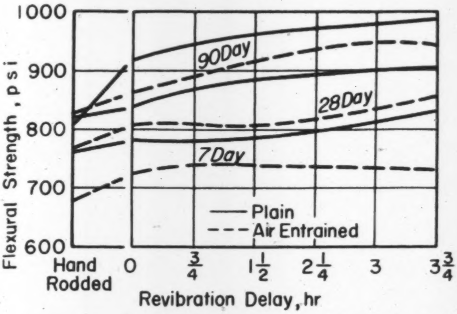

FIG. 4.—Trend Lines Showing Relationship Between Flexural Strength and Revibration Delay for Concrete With and Without Air Entrainment.

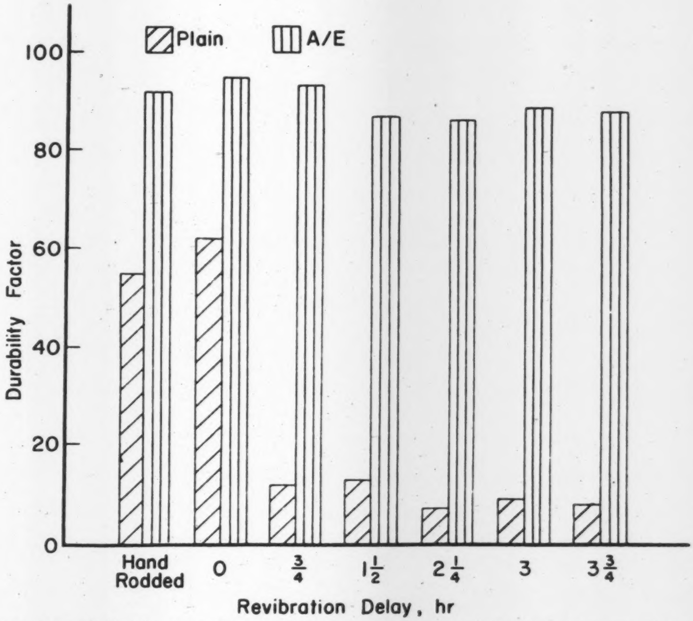

FIG. 5.—Graphical Illustration of Durability Factors After Various Delays of Revibration for Concrete With and Without Air Entrainment.

of the cement and the development of initial hydration products. Any structure arising from hydration products at this point, of course, is governed principally by the C_3A and perhaps to a lesser extent by C_3S.

Flexural Strength:

The trend indicated by the flexural strength data shown in Fig. 4 is similar to that exhibited by the compressive strength specimens but of considerably lesser magnitude. The ultimate increase in flexural strength, above that of specimens given initial vibration only, was approximately 5.2 per cent for the 7-day specimens without air entrainment. This compares to an increase of 5.9 and 5.3 per cent for the corresponding 28- and 90-day specimens, respectively. The air-entrained concrete showed an increase of 2.8, 3.8, and 2.2 per cent at the 7, 28, and 90-day test periods. All strengths continued to increase for samples revibrated after up to a 4-hr delay, after which time it was no longer possible to revibrate. It should be noted that the depth of the flexural beam molds was relatively slight in comparison to that of the cylinders, thus preventing the influences of revibration on those samples to be fully realized. Although there are perhaps other considerations which also influence the flexural strengths obtained, it should be also noted that the trend in this case serves to further illustrate the extent to which the hydration process can be successfully interrupted.

Durability:

Results of the freezing-and-thawing tests, for samples revibrated after the various delay periods, are shown in Fig. 5. The cycles corresponding to a 40 per cent reduction in dynamic modulus were used in calculating the durability factors. The test continued until the specimens had been subjected to 300 cycles, or until the relative dynamic modulus of elasticity of each specimen had decreased to 60 per cent of the initial modulus.

The advantages generally associated with air entrainment are most evident in revibrated concrete, as shown by the bar graphs. The effects of revibration on both the air content and the subsequent durability of the air-entrained concrete specimens were of apparently little significance. Specimens that were hand rodded, those vibrated only initially, and those revibrated after a $\frac{3}{4}$-hr delay exhibited slightly higher durability factors than those undergoing further delay. The magnitude of this difference, however, was limited to 9.5 per cent.

The data show that for the revibrated non-air-entrained concrete, a severe average decrease of 84 per cent in durability was effected against that undergoing only initial vibration. This very marked decrease in durability for all revibrated non-air-entrained concrete is interpreted as evidence of more critical saturation in the mortar. Here, the mortar itself is considered to reflect the difference in durability of specimens hand rodded and initially vibrated as opposed to that of the revibrated specimens. Since the degree of saturation of the aggregates at the time of mixing was assumed to be the same in all instances, any concomitant loss in durability due to revibration seems attributable to changes produced within the mortar. Thus, disturbing the hydration process appears to result in critical saturation of the non-air-entrained paste. On the other hand, paste which initially contained a suitable air content, even though a portion of the air bubbles had been dissipated through revibration, showed little decrease in durability. The absence of air voids in the non-air-entrained paste, which normally relieve some of the internal stresses, can be said to account in part for the poor durability of these specimens.

These concepts may not, however, fully account for the results obtained, since there was relatively little air in the hand-rodded and initially vibrated specimens without air entrainment. The average durability factor for these specimens was 58 as compared to a value in the vicinity of 10 for the revibrated specimens.

Permeability:

Evaluation of the influence of revibration on the permeability of concrete was undertaken on samples from each of the revibration intervals. The specimens tested were 4-in. cores taken from each end of the beams broken in the flexural strength test. Water permeability tests were made on the cores, using a constant head permeameter. The results from these tests were of such wide variation that they could not be adjudged with confidence. In many cases the variation was greater within specimens taken from the same beam than between specimens subjected to revibration after different intervals of delay. No definite trend could be established for either the air-entrained or non-air-entrained concretes. The permea-

TABLE II.—EFFECTS OF VIBRATION TIME ON UNIT WEIGHT AND COMPRESSIVE STRENGTH.

Length of Vibration Time, sec	Unit Weight of Hardened Concrete, lb per cu ft	Average Compressive Strength, psi
15	153.6	4580
30	155.7	4825
45	154.0	4625
60	154.8	5000
75	153.9	4530
120	154.1	4925

bility test was not run on the concrete containing the retarding additive.

Overvibration:

The purpose of vibrating concrete is to consolidate the mass to the point of eliminating, as far as practicable, the voids within the concrete. With revibrated concrete, this feature is extended and accounts for additional consolidation through the disruption of the initial gel structure. Revibration also allows further compaction by eliminating the voids which result from internal bleeding as the concrete hardens. Also, at the point of stiffening at least some of the mixing water has been used up and converted to a hydration product. The extent to which the void content is reduced depends on the length of the delay before revibration and on the amount of vibration.

To provide a basis for estimating the influence of time of vibration on unit weight and compressive strength, 18 cylinders were cast and revibrated for different periods of duration 3½ hr after they were cast. Thus, after a constant revibration delay of 3½ hr, specimens were vibrated for periods varying from 15 to 120 sec, as noted in Table II. On the basis of these data it appears that the length of vibrating time, or the possibility of overvibrating, is not critical insofar as the compressive strength of the concrete is concerned. Also, visual inspection of these specimens after they were broken indicated that there had been no segregation within the mix.

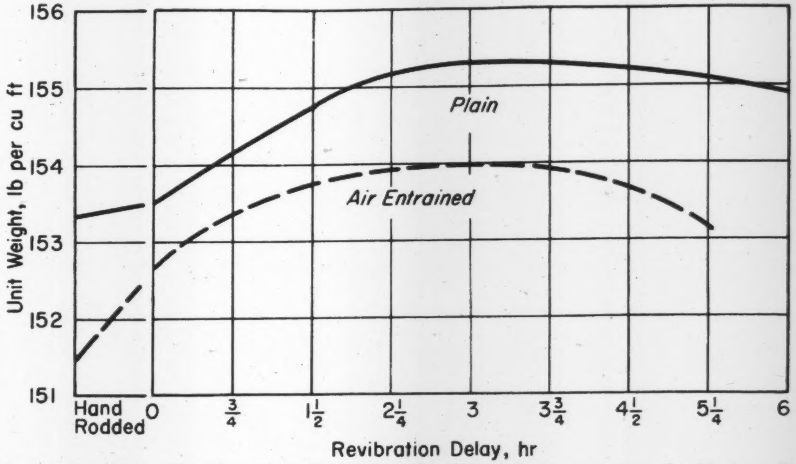

FIG. 6.—Trend Lines Showing Unit Weight of Hardened Concrete after the Various Delays of Revibration.

Unit Weight:

The influence of revibration on the unit weight of concrete for the various intervals of delay is shown in Fig. 6. The unit weight of hardened concrete

TABLE III.—PROPERTIES OF NON-AIR-ENTRAINED CONCRETE AT VARIOUS REVIBRATION DELAYS.

Revibration Interval, hr	Properties of Hardened Concrete														Properties of Fresh Concrete						
	Compressive Strength, psi						Flexural Strength, psi						Average Unit Weight, lb per cu ft		Cement Factor, bag per cu yd	W/C Ratio, gal per bag	Slump, in.	Unit Weight, lb per cu ft		Air Content, per cent	
	7 Day		28 Day		90 Day		7 Day		28 Day		90 Day		Cylinder	Beam				Actual	Calc	Meter	Gravity
Hand Rodded	3510	3680	4730	4710	5575	5995	730	765	820	830	760	790	153.4	153.6	6.01	6.36	3.0	150.4	151.9	1.6	1.0
	3590		5090		5910		800		840		740				6.01	6.36	3.0	150.4	151.9	1.6	1.0
	3950		4320		6500		770		830		880				5.99	6.36	2¾	150.8	150.9	1.8	0.9
Initial Only	3920	3880	5140	5010	6160	6080	920	780	780	830	1020	910	153.5	154.1	6.00	6.07	3¼	150.6	151.2	1.8	1.4
	3860		5115		5880		720		890		920				6.05	5.80	3¼	150.2	153.4	1.9	2.1
	3860		4780		6200		710		810		780				6.05	6.80	3¼	150.2	153.4	1.9	2.1
¾	4290	4370	5790	5810	6960	6790	750	790	780	890	980	970	154.2	154.6	6.00	5.88	2¾	149.4	153.2	1.4	2.5
	4430		5570		6810		810		880		1000				6.00	5.88	2¾	149.4	153.2	1.4	2.5
	4390		6080		6610		800		1020		920				5.99	6.39	2½	150.0	151.8	1.3	1.2
1½	4520	4560	6310	5860	7275	7090	690	750	1000	880	1220	1150	154.7	154.3	6.03	6.00	2¼	150.4	152.9	1.3	1.8
	4345		5710		6800		750		940		1160				6.01	6.00	2½	150.0	152.9	1.3	1.8
	4805		5570		7190		820		690		1070				5.99	6.39	2½	150.0	151.8	1.3	1.2
2¼	4700	4920	5960	5680	6800	7240	720	790	880	880	970	1010	155.2	154.7	6.04	6.26	3.0	151.0	152.1	1.2	0.7
	4750		5650		7260		910		870		990				6.04	6.26	2½	151.0	152.1	1.2	0.7
	5320		5420		7660		730		880		1060				6.00	5.99	2½	150.4	153.0	1.0	1.7
3	4830	5150	6345	6430	7170	7360	860	850	920	850	890	890	155.7	155.3	6.00	5.99	2¾	150.4	153.0	1.1	1.7
	5050		6220		7550		840		620		850				6.00	5.99	2¾	150.3	153.0	1.1	1.8
	5575		6720						1000		940				6.00	5.99	2½	150.4	153.0	1.0	1.7
3¾	5300	5340	6310	6065	7040	7190	800	800	860	940	900	980	155.0	155.0	5.98	5.99	3.0	150.0	153.0	1.0	1.9
	5230				7150		760		980		1020				5.98	5.99	3.0	150.0	153.0	1.0	1.9
	5490		5820		7380		850		980		1020				5.98	5.99	3.0	150.0	153.0	1.0	1.9
4½	5120	5140	5660	6010	7165	7055							154.8		5.98	5.99	3.0	150.0	153.0	1.0	1.9
	4970		6200		6830										6.00	6.00	3½	149.6	152.9	0.9	2.1
	5340		6180		7180										6.00	6.00	3½	149.6	152.9	0.9	2.1
5¼	5190	5090	6090	6250	7040	6980							155.2		6.01	6.00	3½	150.0	152.9	0.9	1.9
	5250		6550		6950										6.01	6.00	3½	150.0	152.9	0.9	1.9
	4840		6120		6950										6.00	6.00	3½	149.6	152.9	0.9	2.1
6	5160	5300	6630	6730	7075	7125							155.2		6.08	5.83	3¼	151.0	153.3	0.9	1.4
	5330		6870		7175										6.08	5.83	3¼	151.0	153.3	0.9	1.4
	5400		6680		...										6.07	5.94	3¾	151.2	153.0	0.9	1.2

TABLE IV.—PROPERTIES OF AIR-ENTRAINED CONCRETE AT VARIOUS REVIBRATION DELAYS.

Revibration Interval, hr	Properties of Hardened Concrete														Properties of Fresh Concrete						
	Compressive Strength, psi						Flexural Strength, psi						Average Unit Weight, lb per cu ft		Cement Factor, bag per cu yd	W/C Ratio, gal per bag	Slump, in.	Unit Weight, lb per cu ft		Air Content, per cent	
	7 Day		28 Day		90 Day		7 Day		28 Day		90 Day		Cylinder	Beam				Actual	Calc.	Meter	Grav
Hand Rodded	3900	3860	4780	4890	6110	5740	660	660	670	770	840	830	151.5	150.6	6.18	5.23	3.0	148.6	157.1	4.6	6.5
	3850		4910		5530		670		840		840				5.87	5.23	2¾	148.2	155.4	4.5	4.6
	3830		4990		5580		640		790		800				5.87	5.23	2¾	148.2	155.4	4.5	4.6
Initial Only	4070	4240	5590	5380	6110	6140	820	770	840	840	900	860	152.7	152.3	5.86	4.89	2½	147.5	156.4	3.9	5.7
	4345		5140		6180		730		850		820				5.86	4.89	2½	147.5	156.4	3.9	5.7
	4310		5400		6140		770		830		860				5.86	4.89	3.0	147.5	156.4	3.9	5.7
¾	4340	4520	5570	5630	6040	6300	780	740	750	770	900	880	153.3	152.2	6.19	5.25	3.0	146.4	155.0	4.5	5.5
	4740		5510		6020		720		740		880				6.19	5.25	3.0	146.4	155.0	4.6	5.5
	4490		5810		6850		710		820		860				6.19	5.25	3.0	146.4	155.0	4.5	5.5
1½	5120	4670	5900	5240	6340	6670	770	700	680	790	840	910	154.1	152.7	5.89	5.45	2½	145.6	154.5	4.3	5.7
	4460		4800		6990		600		800		1080				5.89	5.45	2½	145.6	154.5	4.3	5.7
	4430		5020		6690		840		880		800				5.89	5.65	2¾	145.8	153.9	4.5	5.3
2¼	4610	4780	5390	5750	6200	6650	lost	745	720	760	840	930	153.7	153.8	5.89	5.65	3.0	145.8	153.9	4.5	5.3
	4830		5740		6670		740		940		1060				5.93	5.45	2¾	146.4	153.7	4.3	4.8
	4890		6110		7040		750		620		900				5.93	5.45	2½	146.4	153.7	4.5	4.8
3	5210	5110	6350	6130	6115	6695	600	730	800	860	940	950	154.2	154.4	5.94	5.25	2½	146.4	155.0	4.4	5.5
	5260		6140		7165		640		910		960				5.94	5.25	2½	146.4	155.0	4.4	5.5
	4860		5910		6795		820								5.93	5.25	3.0	146.2	155.0	4.4	5.7
3¾	4660	4730	6190	6180	6970	7050	790	700	840	890	960	940	153.8	154.0	5.93	5.25	3¼	146.2	155.0	4.5	5.7
	5150		6140		6865		680		870		860				5.93	5.25	3¼	146.2	155.0	4.5	5.7
	4370		6220		7310		640		970		1000				5.93	5.25	3¼	146.2	155.0	4.5	5.7
4½	5090	4880	6760	6350	5865	6685	760	780					153.1		5.89	5.40	3.0	145.4	154.6	4.6	5.9
	5070		6230		6540		840								5.89	5.40	3.0	145.4	154.6	4.8	5.9
	4490		6050		7660		730								5.91	5.25	3.0	145.6	155.0	4.8	6.1
5¼	4655	4285	6050	5460	6830								153.5		5.91	5.25	3.0	145.6	155.0	4.8	6.1
	3940		4870												5.95	5.25	3½	146.6	155.0	4.5	5.4
	4260		...												5.95	5.25	3½	146.6	155.0	4.5	5.4

was calculated from the 7-day cylinders revibrated after the different intervals. The maximum unit weight obtained corresponds very nearly to the revibration delays at which the maximum compressive strength occurred. In other words, an increase or decrease in the unit weight parallels that of the compressive strengths obtained. The maximum increase in unit weight for the non-air-entrained concrete occurred in samples revibrated after periods of delay between 2 and 4 hr and was in the order of 1.3 per cent. The peak for the air entrained concrete occurred at about 3 hr. and was approximately 1.0 per cent greater than that also undergoing only *initial vibration*. Any variation in the relationships between the unit weight and compressive strength of a given mix is assumed to indicate that the concrete was not revibrated to the same degree of plasticity in all cases. The maximum unit weights occurred, as did compressive strengths, at about the same time as the initial time of set of the cement (2 hr 45 min) as measured by the ASTM Method of test C 266 - 51 T.[3] Design data and individual strength results for the various mixes are shown in Tables III and IV.

SUMMARY

In disturbing the hardening process of the concrete by revibration, it is, of course, principally the mortar and not the coarse aggregate that is affected. However, there is little reason to believe that tests on the mortar alone would reliably indicate the properties of the concrete, particularly with regard to performance in freezing-and-thawing tests. In order for the strengths to be increased by revibration, either of two conditions would have to be fulfilled: (1) the mortar and concrete would be more densely consolidated, thereby permitting more advantageous deployment of the hydration products, or (2) the vibratory disturbances in some way would accelerate and extend the production and consequently increase the amount of the strengthening hydrates at the particular ages up to 90 days.

The decrease in durabiliy of the revibrated specimens is attributed to changes produced within the mortar or paste rather than the coarse aggregates, since these remained the same in all mixes. While revibration is assumed to bring particles into more intimate contact and thereby reduce the extent to which hydration would normally need to progress in order to achieve satisfactory inter-particle bond, the attending conditions would produce a somewhat denser mortar which would also tend to become more critically saturated by the remaining free water. Thus, the decrease in durability and the increase in strength obtained through revibration may have resulted from the combination of these factors.

The initial structure is determined by either the C_3A or C_3S, whichever hydrates first. In a normally retarded set, the C_3S is presumed to hydrate first and to establish the primary or principal structural bonds. If, on the other hand, the C_3A hydrates first, revibration may displace this weaker structure and allow the normal structure of the C_3S to develop as in an otherwise normally retarded set. The extent of hydration of C_3S is relatively insignificant up to 4 to 6 hr while that of the C_3A may be (unless normally retarded) almost complete within 10 hr. These factors could have significant influence on the rate of strength development as well, and even on ultimate strength.

The apparent gain in strength, or the

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[3] Tentative Method of Test for Time of Setting of Hydraulic Cement by Gillmore Needles (C 266 - 51 T), 1955 Book of ASTM Standards, Part 3, p. 198.

differential between the normally vibrated and revibrated concretes, was most pronounced at 7 days, slightly less at 28 days, and considerably less at 90 days. In other words, the revibrated samples developed strength at a faster rate. While strengths at a 90-day age may not fully reflect ultimate strengths, the 90-day values indicate a more or less persistent or permanent increase attributable to factors other than the rate of hardening.

Conclusions

Although the portions of the study which deal with the effects of revibration on a 4.5-bag mix and on mixes incorporating a retardant have not yet been completed, the data reported and discussed here are considered to be adequate support for the following conclusions:

1. Revibration will increase the strength of concrete after any period of delay as long as the mix can be brought to a plastic state, even if the vibrator must be forced into the concrete.
2. The durability of non-air-entrained concrete is critically decreased, while the durability of air-entrained concrete is not significantly affected by revibration.
3. The possibility of overvibrating after any revibration delay does not appear critical.
4. Revibration at any feasible period of delay does not cause segregation.
5. The optimum time after casting to revibrate occurs when the vibrator will just sink into the concrete of its own accord.

DISCUSSION

Mr. Irwin A. Benjamin.[1]—Was there any relationship between the limiting time the concrete could be revibrated and the initial and final set of the cement as it ordinarily determined?

Mr. D. H. Sawyer (*author*).—The maximum increase in strength was obtained on compression specimens and occurred between 3 and 4 hr. The initial time of set of the cement was around 2 hr and 45 min, which is fairly close to the time that the maximum increase in strength occurred. However, no definite relationships between the time of set and revibration period was established. The vibrator would sink into the concrete of its own accord at any time until the initial setting of the cement occurred, and for this reason the time of initial set could be considered the optimum point at which to revibrate, even though the maximum increase in strength developed after a somewhat greater delay.

Mr. Richard C. Mielenz.[2]—The results indicate that the durability did not decrease or change significantly with the revibration of the air-entrained concrete, whereas the density of the concrete increased very significantly with revibration. This is extremely significant. According to our work, there is every indication that while the concrete is plastic, air is dissolved from the small voids and is released into the larger air voids. This process continues as long as the concrete is plastic.

In my interpretation of the data, with the progressively larger delays of the time of revibration, the large voids increased in size and then with the revibration were released from the concrete but the small voids were not decreased significantly in number or frequency. Consequently, the spacing of the voids remained about the same in spite of a considerable decrease in air content and an increase in density.

Mr. Sawyer.—The air content before vibration for the non-air entrained concrete was approximately one and a half per cent. Upon initial vibration this dropped to about 1 per cent and continued to drop with each delay of the revibration interval. This does indicate that a portion of the air is released upon vibration, and, of course, this would apply to the air entrained concrete as well. However, the fact that revibration produced no significant change in durability for the air entrained concrete and yet greatly decreased the durability of the non-air entrained would indicate that the smaller voids were not significantly affected.

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[1] Director of Research, Granite Steel Co., and Granite Steel Products Co., St. Louis, Mo.

[2] Formerly, Head of Petrographic Lab., Bureau of Reclamation, Denver, Colo., Currently, Director of Research, The Master Builders Co., Cleveland, Ohio.

AUTOMATIC EQUIPMENT AND COMPARATIVE TEST RESULTS FOR THE FOUR ASTM FREEZING-AND-THAWING METHODS FOR CONCRETE*

By H. T. Arni,[1] B. E. Foster,[1] and R. A. Clevenger[1]

Synopsis

A description of automatic apparatus used to operate simultaneously all four of the ASTM tentative methods of test for resistance of concrete specimens to freezing and thawing is given. Details of the development and operation of the apparatus are given together with the problems dealt with in meeting the specification requirements. Time-temperature curves obtained with thermocouples embedded in specimens illustrate the operation of each of the four methods. Data obtained from tests on six concretes made from three different aggregates and with two air contents are given. Confidence intervals are plotted to show which methods best separate the different concretes as well as how many cycles of test are required to give a reliable separation between these concretes.

The main freezing-and-thawing apparatus described herein was designed to provide equipment in which tempertures could be accurately maintained at any point between −10 and 70 F, in which the rate of cooling or heating could be readily adjusted, and in which a number of different conditions could be maintained at the same time.

The general scheme of a reservoir of cold liquid together with ten individual tanks for treating specimens, each controlled independently of the others, was adapted from the previous National Bureau of Standards equipment described in the 1946 ASTM Symposium on Freezing-and-Thawing Tests of Concrete (**1**).[2] The geometrical arrangement of the tanks and piping system was modified as the result of operating experience obtained with the previous equipment.

As long as the temperature of the reservoir in such a system remains practically constant, the treatment to which specimens are subjected is independent of the operating efficiency of the refrigeration machines. This is in contrast to many installations where the freezing rate is determined by the capacity of the refrigeration condenser and evaporator, a capacity which may change appreciably with valve and cylinder wear, gas charge, condensing water temperature, and expansion valve operation. This advantage is obtained, however, at a sacrifice

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] Materials Engineer, National Bureau of Standards, Washington, D. C.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1253.

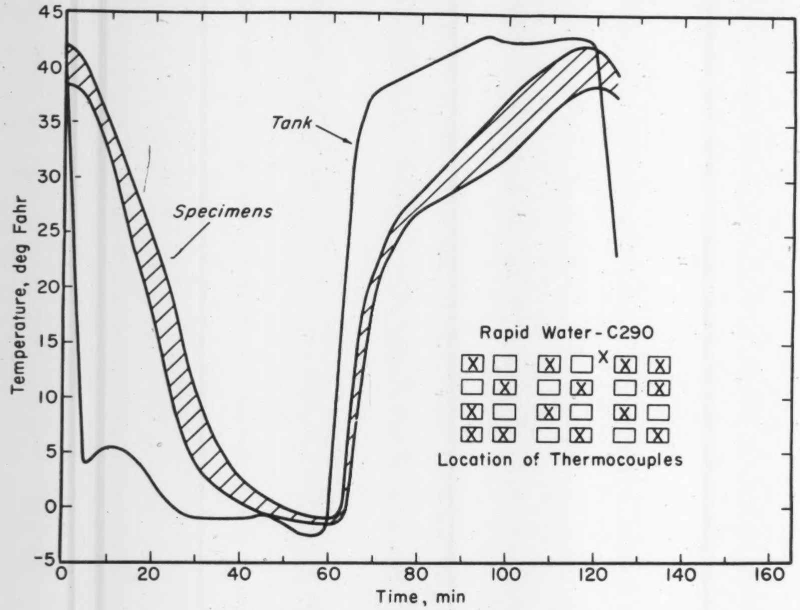

FIG. 1.—Rapid-Water Performance Graph.

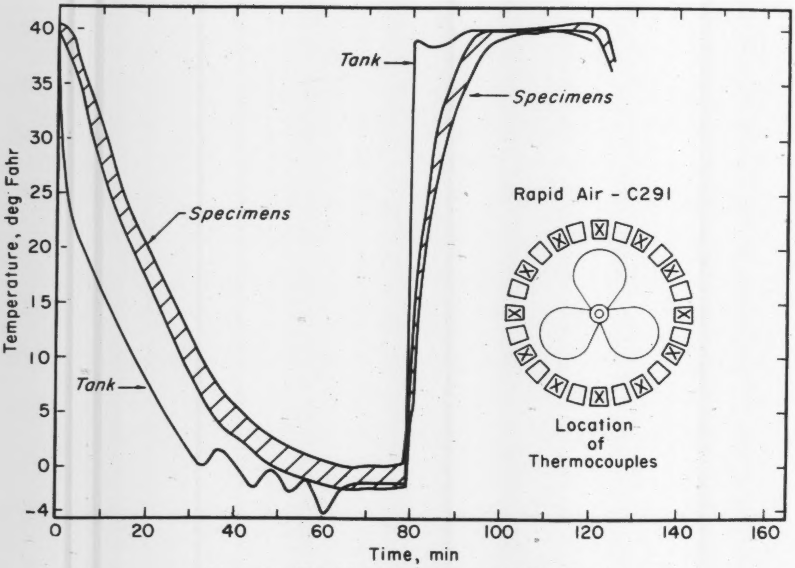

FIG. 2.—Rapid-Air Performance Graph.

in refrigeration capacity due to the extensive pumping, piping, and tank system with their attendant heat gain. Also, the refrigeration system is removing heat at a low temperature, a condition in which its capacity is reduced as compared with operation at a higher temperature. It was felt, however, that for the work in mind, the advantages of the system as designed outweighed the disadvantages.

During construction, the desirability of adding automatic equipment similar to that employed by the Corps of Engineers (2) and the Bureau of Reclamation (3) for use in conducting routine tests on aggregates was recognized. As a result two large treatment tanks together with reservoirs for storing freezing-and-thawing liquids were added. Later, with the adoption by the ASTM of four tentative standard freezing-and-thawing procedures, it became apparent that the comparison of the durability of various concretes as subjected to each of the four types of exposure would be of value. The automatic equipment mentioned above is designed to meet the requirements of ASTM Method C 290 - 52 T, Rapid Freezing and Thawing in Water,[3] while the requirement of Method C 292 - 52 T for Slow Freezing and Thawing in Water[4] may be met in six of the small individual tanks.

To round out the equipment, two cold boxes, previously used for other purposes were modified to comply with the requirements of ASTM Method C 291 - 52 T[5] and Method C 310 - 53 T.[6] Rapid

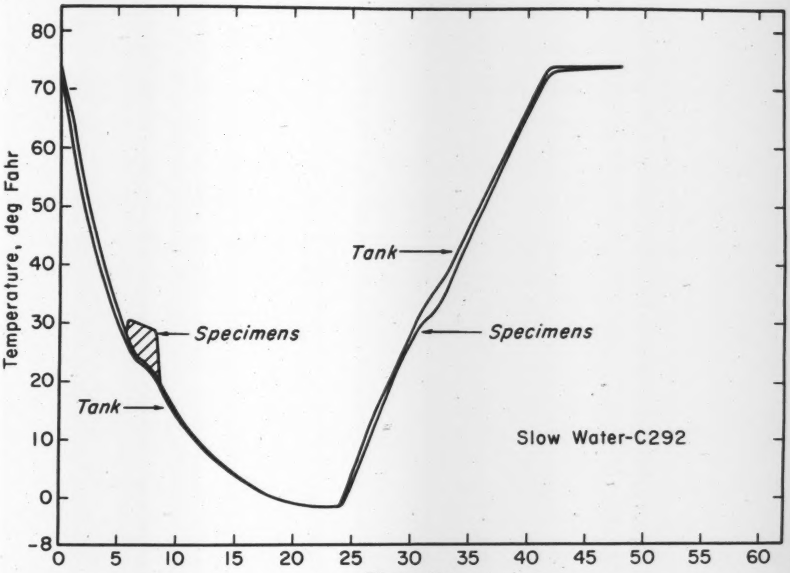

Fig. 3.—Slow-Water Performance Graph.

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[3] Tentative Method of Test for Resistance of Concrete Specimens to Rapid Freezing and Thawing in Water (C 290 - 52 T), 1955 Book of ASTM Standards, Part 3, p. 1339.

[4] Tentative Method of Test for Resistance of Concrete Specimens to Slow Freezing and Thawing in Water or Brine (C 292 - 52 T), 1955 Book of ASTM Standards, Part 3, p. 1347.

[5] Tentative Method of Test for Resistance of Concrete Specimens to Rapid Freezing in Air and Thawing in Water (C 291 - 52 T), 1955 Book of ASTM Standards, Part 3, p. 1343.

[6] Tentative Method of Test for Resistance of Concrete Specimens to Slow Freezing in Air and Thawing in Water (C 310 - 53 T), 1955 Book of ASTM Standards, Part 3, p. 1351.

Freezing in Air and Thawing in Water and Slow Freezing in Air and Thawing in Water, respectively. Operation of these is similar to that of the apparatus of the National Sand and Gravel Association (4).

Since thawing takes place in water in all four types of test, Methods C 290, C 291, C 292, and C 310 will henceforth be referred to herein as rapid-water, rapid-air, slow-water, and slow-air methods or systems respectively.

Curves are presented showing the performance of the apparatus as used in the four ASTM freezing-and-thawing procedures.[3, 4, 5, 6] Also data are given on a comparative program in which six different concretes were tested by each of the four methods. A description of the various pieces of apparatus is presented in the Appendix.

PERFORMANCE TESTS OF EQUIPMENT

Graphs of the temperature changes in the system, both ambient and at the centers of the specimens, are shown in Figs. 1, 2, 3, and 4, each graph portraying a typical cycle of operation of the apparatus. These temperature readings were obtained with copper-constantan thermocouples, the thermocouples for specimen temperatures being embedded in the centers of 3 by 4 by 16-in. "dummy" concrete beams similar to the test specimens (see Appendix). Each system meets the requirements of the applicable ASTM method except as noted.

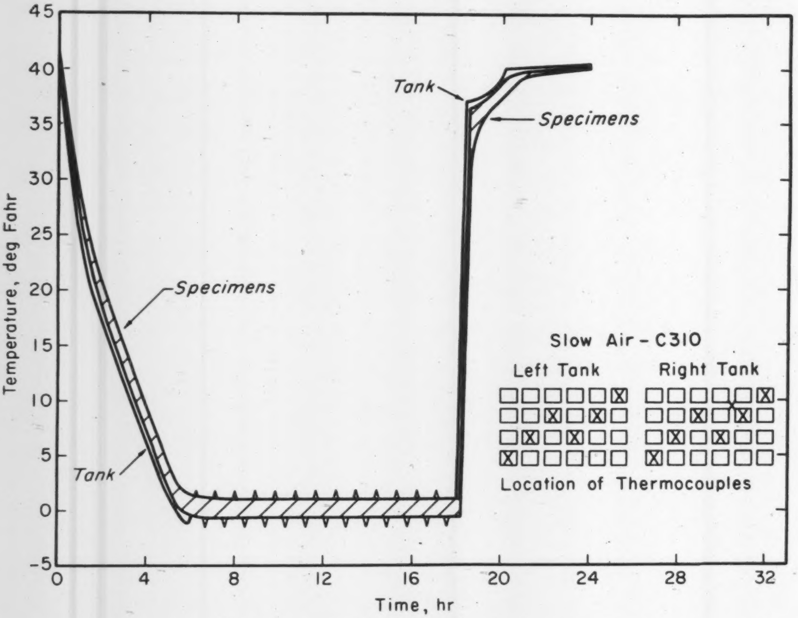

FIG. 4.—Slow-Air Performance Graph.

Rapid-Water System:

The rapid-water graph (Fig. 1) shows the range of temperatures indicated by 14 thermocouples distributed throughout one specimen tank as shown. In this system the specimens are placed upright in the treatment tank in close-fitting copper cans and are covered with equal amounts of water.

Each specimen either contained a thermocouple or was adjacent to two others which contained thermocouples. The tank temperature was obtained from a single thermocouple between the racks as shown. Conditions in both tanks are the same. All of these thermocouples were at the center of the specimens. Previous tests with thermocouples near the top and bottom and at the center of specimens have indicated that treatment throughout the length of the specimens is essentially the same.

The curve indicates failure to meet the specification requirement that "the time required to change the temperature at the center of the specimens shall not vary . . . more than 10 min when changing from 0 to 40 F."[3] The slower specimens reach only 38.5 F at the end of the thawing period, but the faster specimens reach this temperature about 16 min sooner. The requirement is difficult to meet due to the fact that the end thawing temperature is not very far above the thawing temperature of the ice in the cans and there is usually some ice left around the top of the specimens. The volume of the cans was adjusted at the beginning so that they contained approximately the same amount of water (see Appendix). However, after a few cycles, specimens which do not fail early in this system begin to slough off drastically and may lose as much as 10 to 20 g per cycle. This makes it impossible to keep a constant volume of water in the cans, and it has sometimes been necessary to gradually raise the temperature of the thawing alcohol almost to 50 F to melt the ice on the sides of the specimens as tests on a given group of specimens progressed.

Rapid-Air System:

The graph of the rapid-air system is shown in Fig. 2. The curve for the specimens was obtained with 24 specimens arranged in a ring in the tank and with 12 thermocouples, one in every other specimen as shown. The tank temperature was obtained with a single thermocouple inside the ring of specimens at about the middle height.

Thawing is extremely rapid in this system since the ratio of thawing water volume to specimen volume is large. Also the large refrigeration capacity (approximately 25 tons) and heavy circulation of air produce a rapid freezing rate.

Slow-Water System:

The slow-water system curve is shown in Fig. 3. This curve was obtained from only one of the six individual tanks which are adapted for this system. However, the tanks are identical and the cooling and heating rates are easily adjustable so that identical curves were obtained from each of the six tanks. The specimen curve shown was obtained with eight thermocouples, one in each of the eight specimens in the tank. The specimens were in rubber boots with sufficient tap water to cover the specimen in each boot. All of the slow-water freezing is done in water rather than in brine, a choice allowed by the specification[4]. The specimens are placed in the boots in the racks outside the tank and then the rack is placed in the tank. Each boot is then filled with sufficient water to just cover the specimen and is manipulated by hand to eliminate all air trapped around or under the specimen. The tops of the boots are then folded over brass rods and clamped so that the boots are closed and the specimens are supported in a vertical position in the rack. The ambient temperature was obtained from one thermocouple taped to an upright of the rack.

TABLE I.—COMPOSITION OF AGGREGATES.

Aggregate	Composition	Approximate per cent
A........	Almost entirely quartz and quartzite pebbles, rounded and well-polished	...
B........	Quartz and quartzite Dense chert Soft, porous chert Miscellaneous (sandstone, granite, hematite, limonite)	77 11 9 3
C........	Dense, hard chert Soft, porous chert Quartz and quartzite Miscellaneous (wood, cinders, shell)	71 26 2.5 2.5
Sand.....	Quartz grains with small amounts of mica and iron oxide	...

TABLE II.—CHARACTERISTICS OF AGGREGATES.

	ASTM Method	Aggregate A	Aggregate B	Aggregate C
Specific gravity	C 127[a]	....	....	....
SS dry	......	2.64	2.58	2.38
Absorption, per cent	C 127[a]	....	....	....
Vacuum saturated	......	0.3	1.4	6.9
24-hr soaked	......	0.3	1.4	4.5
Magnesium sulfate soundness	C 88[b]	....	....	....
5 cycles, per cent loss	......	0.2	5.8	2.5
L. A. Abrasion	C 131[c]	....	....	....
Wear, per cent	......	41	40	22

[a] Method of Test for Specific Gravity and Absorption of Coarse Aggregate (C 127 – 42), 1955 Book of ASTM Standards, Part 3, p. 1231.

[b] Tentative Method of Test for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (C 88 – 55 T), 1955 Book of ASTM Standards, Part 3, p. 1225.

[c] Method of Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine, (C 131 – 55), 1955 Book of ASTM Standards, Part 3, p. 1188.

Due to the slow rate of cooling in this method, the specimen temperatures and the tank temperature are very close together throughout the cycle except the points where freezing and melting of the water occurs. The eight specimen temperatures were so close together that they form one line on the graph except at the freezing temperature. From 32 F to about 28 F supercooling occurs, and when freezing starts the specimen temperature goes up due to the heat released by the freezing water. This phenomenon did not occur at the same temperature for all specimens, and the loop shown in the curve is the envelope of the specimen temperatures.

Slow-Air System:

Figure 4 shows a graph of the slow-air system performance obtained from twelve specimen thermocouples and one unconfined thermocouple distributed as shown on the diagram. This curve indicates conformity with the specification with regard to freezing and thawing time and temperatures, length of cycle, etc., but the range of freezing and thawing time among specimens is outside the limits.[6] During the freezing phase, the slower specimens reach 2 F ½ hr later than the faster freezing specimens. Also, during the thawing phase, there is a spread of approximately ¾ hr between the times the slower and faster specimens require to reach 39 F. This agrees with curves obtained by other laboratories using this type of cycle, and it appears that the specification limits of 20 min between specimens for freezing and 10 min for thawing may be unrealistic, this close a tolerance being difficult to meet.

COMPARATIVE TEST PROGRAM

The program described below was undertaken with the object of comparing the relative ability of the four ASTM freezing-and-thawing methods to discriminate between concretes made with different aggregates. Six concretes were made from three aggregates. Data on these concretes and on the aggregates are given in Tables I, II, III, IV, and V.

Materials:

The concretes were made and tested in two series. In series I, consisting of concretes I A, I B, and I C (Table V), the coarse aggregates were vacuum-saturated. Correct amounts of each size fraction for one batch were combined in a canvas bag and placed in a large air-tight tank. The tank was then clamped shut and pumped down to less than 5

TABLE III.—COARSE AGGREGATE GRADING, ASTM METHOD C 136.[a]

Retained on	Cumulative per cent
1-in. sieve	0
¾-in. sieve	10
½-in. sieve	45
⅜-in. sieve	72
No. 4 sieve	100

[a] Method of Test for Sieve Analysis of Fine and Coarse Aggregates (C 136 – 46), 1955 Book of ASTM Standards, Part 3, p. 1218.

TABLE IV.—FINE AGGREGATE GRADING, ASTM METHOD C 136.[a]

Retained on	Cumulative per cent
No. 4 sieve	0
No. 8 sieve	17
No. 16 sieve	35
No. 30 sieve	55
No. 50 sieve	80
No. 100 sieve	94
Passing No. 100 sieve	6

[a] Method of Test for Sieve Analysis of Fine and Coarse Aggregates, (C 136 – 46), 1955 Book of ASTM Standards, Part 3, p. 1218.

TABLE V.—DATA ON CONCRETES.

Concrete	Aggregate	Aggregate Treatment	Mix Proportions, by weight, per cent			Cement Factor, bags per cu yd	Water-Cement Ratio	C 231[a] per cent air by meter	C 143[b] Slump, in.	C 39[c] 28-day Compressive Strength, psi
			Cement	Sand	Gravel					
I A	A	Vac. Sat.	1	2.78	3.33	5.5	0.60	1.2 to 1.6	2⅝ to 4⅛	3860 to 3970
I B	B	Vac. Sat.	1	2.78	3.19	5.5	0.60	1.5 to 2.0	2½ to 3½	3670 to 3900
I C	C	Vac. Sat.	1	2.78	3.07	5.5	0.58	1.5 to 1.9	1½ to 2	3660 to 3850
II A	A	Soaked 24 hr	1	2.45	3.33	5.5	0.49	6.8 to 8.0	2 to 3⅛	3550 to 4220
II B	B	Soaked 24 hr	1	2.45	3.25	5.5	0.49	6.3 to 8.0	2⅞ to 4¼	3140 to 3590
II C	C	Soaked 24 hr	1	2.45	3.00	5.5	0.49	6.7 to 8.2	1⅝ to 3⅛	3040 to 4000

[a] Method of Test for Air Content of Freshly Mixed Concrete by the Pressure Method, (C 231 – 54), 1955 Book of ASTM Standards, Part 3, p. 1287.

[b] Method of Test for Slump of Portland-Cement Concrete, (C 143 – 52), 1955 Book of ASTM Standards), Part 3, p. 1370.

[c] Method of Test for Compressive Strength of Molded Concrete Cylinders (C 39 – 49), 1955 Book of ASTM Standards, Part 3, p. 1312.

mm of mercury pressure. Minimum pressure was obtained in about 1 hr and the aggregates were left in the tank with the vacuum pump running overnight. In the morning the pump was cut off and water admitted to break the vacuum and inundate the aggregates. The batches were left in the water one day. Aggregate for nine batches of concrete could be saturated at one time in this manner. No air-entraining agent was added to the mixes made with these aggregates.

The vacuum-saturation treatment made little difference in the measured absorption of gravels A and B, but aggregate C, which contained 26 per cent of soft, porous chert, showed an increase in measured absorption from 4.5 to 6.9 per cent.

TABLE VI.—CHARACTERISTICS OF CEMENT.

Characteristics		ASTM Method
Chemical:		
Magnesium oxide (MgO), per cent	1.6	C 114[a]
Sulfur trioxide (SO_3), per cent	1.7	C 114[a]
Total alkali as Na_2O, per cent	0.26	C 114[a]
Tricalcium silicate ($3CaO \cdot SiO_3$), per cent	48	C 114[a]
Tricalcium aluminate ($3CaO \cdot Al_2O_3$), per cent	6	C 114[a]
Physical:		
Specific surface, air permeability, sq cm per g	3400	C 204[b]
Air content of mortar, per cent	6.3	C 185[c]
Compressive strength, psi:		C 109[d]
3-day	1490	
7-day	2280	

[a] Methods of Chemical Analysis of Portland-Cement (C 114 – 53), 1955 Book of ASTM Standards, Part 3, p. 69.

[b] Method of Test for Fineness of Portland Cement by Air Permeability Apparatus (C 204 – 55), 1955 Book of ASTM Standards, Part 3, p. 145.

[c] Tentative Method of Test for Air Content of Hydraulic Cement Mortar (C 185 – 55 T), 1955 Book of ASTM Standards, Part 3, p. 43.

[d] Tentative Method of Test for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. Cube Specimens) (C 109 – 54 T), 1955 Book of ASTM Standards, Part 3, p. 129.

In the series II tests (concretes II A, II B, and II C, Table V), the aggregate batches were merely inundated with water for 24 hr. Also, sufficient neutralized vinsol resin was added to the mixes to raise the air content to 6 to 8 per cent.

The fine aggregate was sand from the same source as coarse aggregate A and was merely soaked 24 hr for all six concretes.

The cement used was a type-II, low-alkali cement meeting all the ASTM requirements for that type. The same cement was used for all mixes. Chemical and physical characteristics are given in Table VI.

Mixing and Curing:

The concrete was mixed in 0.9 cu-ft batches in a tilting-drum type mixer. At the beginning of each day's run, a "butter" batch was made and discarded, then six regular batches were made, four with one aggregate and one with each of the other aggregates.

The weighed aggregates and water were placed in the mixer and mixed 2 min. Then the cement was added and mixing continued for 2 min. The mixer was stopped and wet burlap placed over the opening for a 3-min waiting period. Then the batch was again mixed for 1 min.

The mix was then discharged into a pan and the unit weight and gravimetric air content determined according to ASTM Method C 138.[7] The material from the unit-weight determination was returned to the pan, the entire lot remixed with a shovel, and the slump and air content by air-pressure meter determined. The air meter determinations were generally higher than the gravimetric. The average difference was about 0.4 per cent with the greatest difference for any mix being 1.1 per cent. Three bars, 3 by 4 by 16 in. were cast from the remaining material. The 6 by 12-in. cylinders for compressive strength determinations were cast from the slump material.

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[7] Method of Test for Weight per Cubic Foot, Yield and Air Content (Gravimetric) of Concrete, (C 138 – 44), 1955 Book of ASTM Standards, Part 3, p. 1382.

The specimens were stored in the molds in the fog room at 73 ± 2 F for 48 hr. Then they were stripped, weighed, and replaced in the fog room for 12 days. Freezing-and-thawing tests were started at 14 days.[8]

Freezing-and-Thawing Tests:

On the fourteenth day the specimens for test in all except the slow-water system were put to soak in water at 40 F for approximately 1 hr. The specimens were then weighed, tested for fundamental transverse frequency by vibration in the 3-in. direction in accordance with ASTM Method C 215[9] and placed in the freezing apparatus. Specimens for the slow-water system were measured at room temperature.

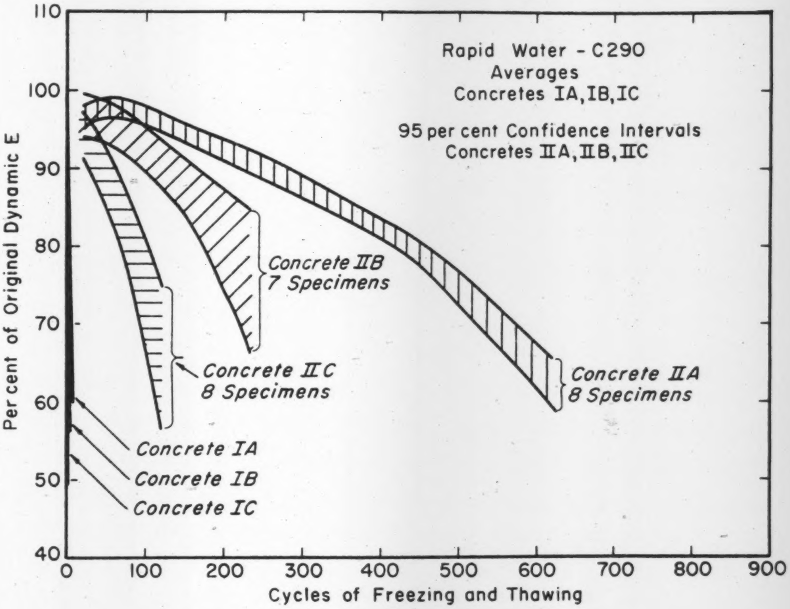

FIG. 5.—Rapid-Water Specimens Graph.

It was originally planned to test eight specimens of each concrete in each freezing-and-thawing system at the same time, but the rapid-air apparatus was not finished when the series I specimens were ready for test. When the tests and adjustments of the rapid-air apparatus were completed, two more rounds (twelve mixes) of specimens were cast and half

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[8] Most of the specimens were removed from the fog room on the thirteenth day and a 2-in. piece was sawed from the end of each one. The cut ends were saved for possible future microscopic determinations of air content. There was no significant difference in performance in the freezing-and-thawing tests between the specimens which were cut and those which were not.

[9] Tentative Method of Test for Fundamental Transverse, Longitudinal, and Torsional Frequencies of Concrete Specimens, (C 215 – 55 T), 1955 Book of ASTM Standards, Part 3, p. 1355.

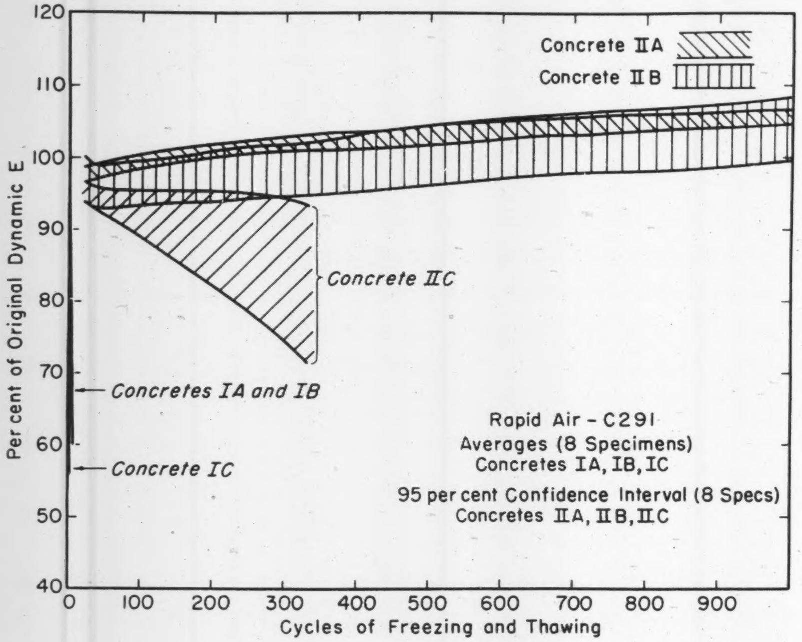

FIG. 6.—Rapid-Air Specimens Graph.

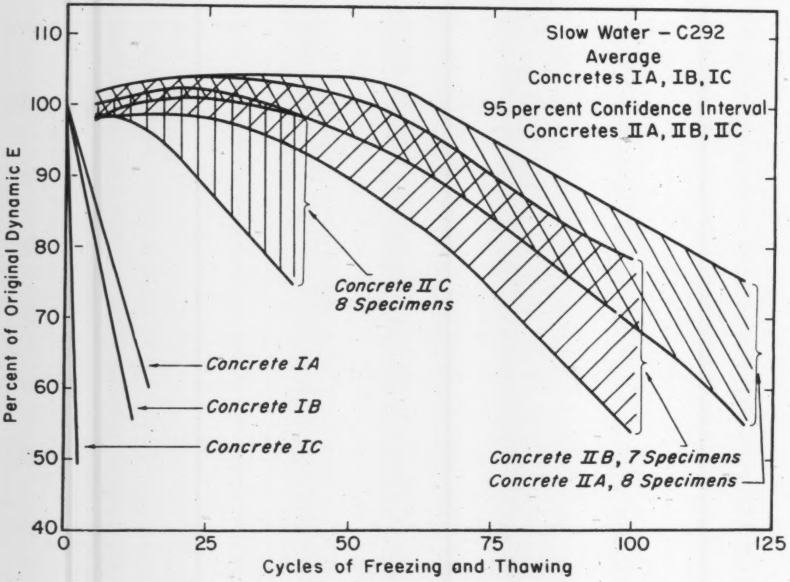

FIG. 7.—Slow-Water Specimens Graph.

were tested in the rapid-air system and half were tested in the rapid-water system to give a comparison between the earlier runs and the rapid-air tests. There was no significant difference between the control specimens tested at this time and the earlier tests.

The series II concretes were tested in all four systems at the same time as originally planned.

At the start of each test, specimens were weighed and tested by the sonic method at intervals of one to five cycles. Specimens whose modulus decreased rapidly continued to be tested at frequent intervals. The more durable specimens were tested less frequently as the number of cycles increased. From the fundamental transverse frequency and the measured weight, the dynamic modulus of elasticity and its ratio to the original dynamic modulus expressed as per cent (hereafter called relative modulus) were calculated using a constant value for C, the constant determined from the dimensions of the specimen.[9] Durability factors were computed at 60 per cent of original dynamic modulus or 300 cycles of freezing and thawing, whichever occurred first. Some of the specimens were continued in the test beyond 300 cycles, however, and the series II bars are still running in the slow-air system at more than 900 cycles with no loss of dynamic modulus (except for one specimen of concrete II C). Some specimens were continued in the rapid-air system to over 1300 cycles without suffering any reduction in dynamic modulus.

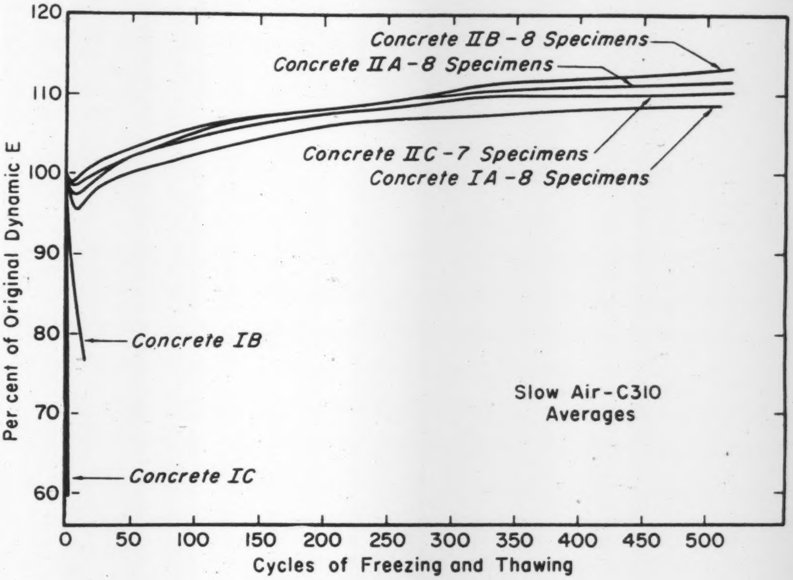

FIG. 8.—Slow-Air Specimens Graph.

TEST RESULTS AND DISCUSSION

Figures 5 to 8 show graphs of the performance of the six concretes under each of the four methods of test.

Confidence Intervals:

The curves shown on the four graphs were obtained in the following manner. As the tests progressed and sonic readings were taken on the specimens, the relative dynamic modulus for each specimen was plotted on a graph for the particular method and concrete. These plots were joined by broken lines, making a family of eight curves for each concrete in each method. At intervals along the abscissa of each graph (where the specimens lasted long enough to make this feasible) the average of the relative moduli of the eight specimens was calculated, and from this the standard deviation and 95 per cent confidence limits for the mean were computed.[10]

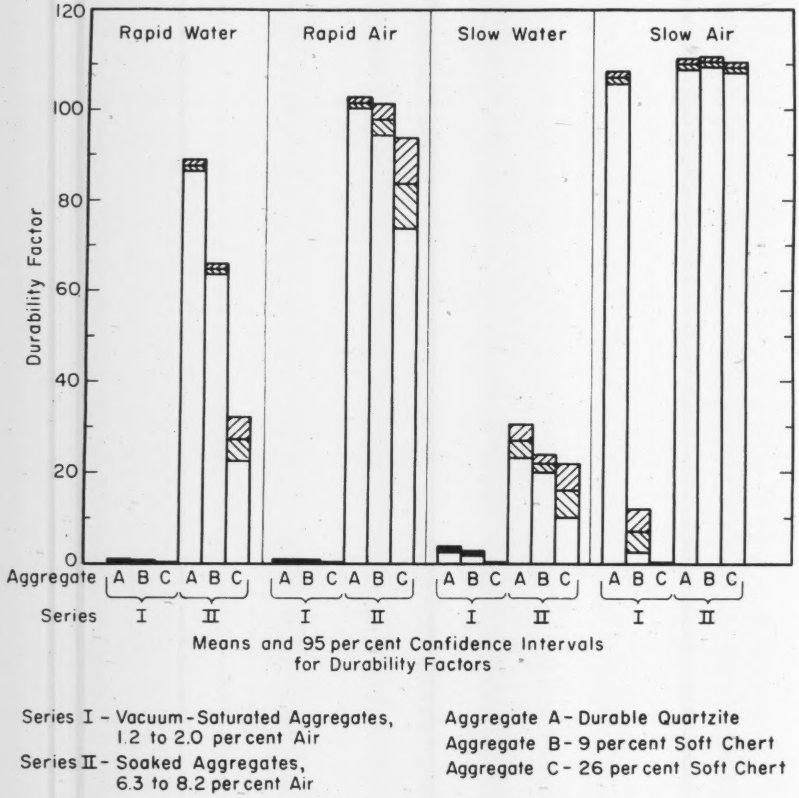

FIG. 9.—Bar Graph of Average Durability Factors.

In Figs. 5 to 7, the curves for the series II concretes are the confidence limits plotted above and below the mean for each group of specimens. The mean lines themselves are not shown, but if they were drawn, they would appear half way between the corresponding confidence limits. Where the upper confidence level for one group separates from the lower level for another group, it can be assumed that the test method has discriminated between the two types of concrete at approximately the number of freezing-and-thawing cycles where this occurs. The series I concretes in these three methods failed so rapidly that no confidence limits could be shown on the graphs, therefore only a line indicating the average of the specimens is shown.

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[10] The 95 per cent confidence limit is an estimate of the limits of the region on either side of the mean of the eight (or seven) specimens within which the true mean (of the population from which these specimens are drawn) would lie 95 per cent of the time. This region is called the confidence interval. It is calculated from the following formulas:

For 8 specimens:

95 per cent confidence limit = $\frac{2.36}{\sqrt{8}}\sigma$

For 7 specimens:

95 per cent confidence limit = $\frac{2.45}{\sqrt{7}}\sigma$

where σ is the standard deviation of the specimens ((**5**), p. 108).

In Fig. 8, the slow-air method, only the average lines are shown for all six concretes. Concretes I B and I C failed too rapidly to permit calculation of limits, and the other four followed so nearly the same curve that the confidence limits for all of them overlapped for the duration of the test.

All of the curves represent eight specimens except concrete II B—rapid-water (Fig. 5), concrete II B—slow-water (Fig. 7), and concrete II C—slow air (Fig. 8). In each of these cases one specimen which failed much earlier than the others was eliminated according to the following statistical criterion (5, p. 243). If the specimens are ranked from the highest to the lowest relative dynamic *E*, and the eighth specimen differs from the seventh by half the difference between the eighth and the second, the eighth is eliminated.

Since the means of calculating both an average and its confidence limits is lost when one specimen fails, some of the curves (notably concrete II C in the rapid-air and slow-water methods) are cut off well above the 60 per cent relative *E* which is defined as failure, although all of the specimens in these instances were continued in the test to failure. In order to present the complete information from all of the specimens, the average durability factors and their 95 per cent confidence limits (the cross-hatched areas at the tops of the bars) are plotted as vertical bars for each concrete in each method in Fig. 9. The bars are based on seven instead of eight specimens in the same three cases as on the graphs, and failure or success in differentiating between two concretes in a given method is indicated by overlapping or absence of overlapping of the confidence intervals for those concretes.

Series I Concretes:

The series I concretes failed so quickly in both the rapid systems, due to the combination of vacuum-saturated aggregate and mortar unprotected by entrained air, that very little information was obtained from those two methods. The only definite conclusion that can be drawn is that the series I concretes are distinctly separated from the series II concretes (Figs. 5, 6, and 9). The order of durability of the three concretes of Series I appears to be the same in both rapid methods, with concrete I A lasting longest and concrete I C failing first, but the difference is not significant.

In the slow-water system the apparent separation of the three curves on the graph (Fig. 7) is not significant since the confidence limits above and below the curve are so wide. When the durability factors, which include information on all the specimens out to failure, are compared, however (Fig. 9), concrete I C is seen to be significantly separated from both concretes I A and I B. Here again all the concretes of series I are distinctly separated from the concretes of series II.

In the slow-air system, the series I concretes are all separated from each other. The separation between concretes I B and I C is not evident from the graph (Fig. 8) but is shown by the comparison of the durability factors. The series II concretes are clearly separated from concrete I B and I C but not at all from concrete I A.

One main fact brought out by the series I concretes is the extreme mildness of the slow-air method. Here concrete I A, which failed in 3, 4, and 15 cycles, respectively, in the other three methods, continued gaining strength far beyond the maximum of 300 cycles recommended[6] and showed no evidence of damage whatsoever, in spite of the fact that the paste was not protected by entrained air. The specimens of this group which were started earliest continued under test for 20 months.

Series II Concretes:

Significant separations between the series II concretes are tabulated in Table VII. The rapid-water method is the only one which discriminated among all three of the concretes. These separations were early and clear cut.

TABLE VII.—NUMBER OF FREEZING-AND-THAWING CYCLES TO PRODUCE SIGNIFICANT SEPARATION FOR SERIES II CONCRETES.

Concretes	Rapid-Water	Rapid-Air	Slow-Water	Slow-Air
II A–II B	100	No Sep.	No Sep.	No Sep.
II A–II C	30	20	Doubtful	No Sep.
II B–II C	45	300	No Sep.	No Sep.

In the rapid-air system there is no separation between concretes II A and II B at all, the limits for II A being included within the limits for II B from about 400 cycles on. The separation between concretes II A and II C is quite decisive and that between concretes II B and II C is borderline, both on the curve and on the bar graph.

Separation between concretes II C and II A appears to be occurring at the end of the II C curve in the slow-water system. This is insufficient evidence for saying that a separation has taken place, but in Fig. 9 a significant separation is shown. In the other two cases in this method, the confidence intervals overlap throughout.

As far as evidence from the slow-air system is concerned, there is no indication of any difference between these three concretes, in spite of the differences in the character of the aggregates from which they were made.

Comparison of Freezing-and-Thawing Methods:

A comparison of the results obtained by the four methods as shown in Fig. 9 indicates that for these six concretes and the apparatus used in this investigation, the rapid-water method gave the best discrimination between the series II concretes and the slow-air method between the series I concretes. The best discrimination between the three aggregates was obtained with series II in the rapid-water method. Here the mortar was protected by the entrained air and the relative durability of the three aggregates shows up very clearly.

In all cases where there was a separation between the three aggregates, whether statistically significant or only apparent, the order of durabilities was the same, with the *A* aggregate most durable and the *C* aggregate least durable. This is, of course, the order that would be expected from the character of the aggregates.

In general, the slow-water method caused the most disintegration per cycle. Due to the slowness of the method, however, failure required a longer elapsed time than in the rapid-water method. For concrete II A, for instance, in the slow-water system the average number of cycles for failure was 135 which required 270 days. In the rapid-water system, all the concrete-II A specimens ran over 300 cycles, but they reached this figure in approximately 60 days.

Separation of Specimens Within a Given Concrete:

An interesting phenomenon which appears to be related to non-homogeneity of the aggregates occurred in concretes II B and II C in both rapid methods of test. The data from these concretes in these methods showed two well-defined groups except for concrete II C in the rapid-air system in which three groups appeared. The average durability factors and numbers of specimens involved in each case are given in Table VIII. The application of a "*t*" test showed all of these differences to be significant[11]. This behavior of these two concretes, in which the aggregates contained approximately 9 per cent and 26 per cent respectively of porous chert, might be explained by the presence or absence of large pieces of the non-durable aggregate in critical locations in some specimens and not in others.

Concretes I B and I C failed so rapidly in all four methods that there was no opportunity for groups to appear. This may also have been the reason for the lack of separation into groups in the case of concretes II B and II C in the slow-water method. In the slow-air method the concretes were so durable that there was no more chance for separation between groups within a concrete than for separation between the different concretes. The A aggregate, of course, was quite uniformly durable and showed no groups in any method in either of the concretes made from it.

TABLE VIII.—GROUPS WITHIN A GIVEN CONCRETE.

	Rapid-Water Method				Rapid-Air Method			
	Concrete II B		Concrete II C		Concrete II B		Concrete II C	
	Avg. D.F.[a]	Number of Specimens	Avg. D.F.[a]	Number of Specimens	Avg. D.F.[a]	Number of Specimens	Avg. D.F.[a]	Number of Specimens
Group 1	75.0	4	33.8	3	101.2	5	97.0	2
Group 2	51.2	3	23.4	5	93.3	3	86.1	4
Group 3	...	...	...	...	...	...	66.8	2

[a] Average durability factors.

SUMMARY AND CONCLUSIONS

A test program is described in which six concretes were studied simultaneously by all four ASTM methods for testing the resistance of concrete to freezing and thawing. The concretes were made with three aggregates, one durable, one non-durable, and one intermediate, one series being made with vacuum-saturated aggregates and no purposefully-entrained air, and a second series with soaked aggregates and 7 per cent air. Averages and confidence intervals representing the performance of the six concretes in each method are given.

Time-temperature curves are given representing the performance of the four systems for conducting the tests, and some of the problems encountered in meeting the requirements of the methods are discussed. The development and operation of the automatic apparatus is described in the Appendix.

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[11] The "*t*" test is a statistical test for determining whether the difference between the averages of two groups of data can be regarded as significant, ((**6**), p. 24). The difference between the averages for groups 1 and 2, concrete II C in the rapid-air method was significant at the 5 per cent level. All the others were significant at better than the 0.5 per cent level.

The following conclusions are drawn:

1. With the series I concretes (vacuum-saturated aggregates and no entrained air):

(*a*) Significant discrimination between all three aggregates was obtained only in the slow-air method;

(*b*) In the rapid-water and rapid-air methods, disintegration was too rapid to show any differences between the three aggregates.

(*c*) In the slow-water method, concrete I C was separated from concretes I A and I B.

2. With the series II concretes (aggregates merely soaked and 7 per cent air included):

(*a*) In the rapid-water method aggregates A and B were separated at 100 cycles of freezing and thawing, aggregates A and C at 30 cycles, and aggregates B and C at 45 cycles;

(*b*) In the rapid-air method aggregates A and B were not separated, but aggregates A and C were separated at 20 cycles and aggregates B and C at 300 cycles;

(*c*) In the slow-water method aggregregates A and C were separated at 40 cycles, but aggregate B was not separated from either of the others;

(*d*) In the slow-air method there was no separation.

3. All of the series II concretes were separated from all of the series I concretes in all methods, except that concrete I A was not separated from series II in the slow-air method.

4. For these aggregates and concretes, the rapid-water method gave the best separation for the more durable concretes and the slow-air system gave the best separation for the least durable concretes.

5. Specimens with the most durable aggregate and entrained air (concrete II A) showed no deterioration up to over 1300 cycles in the rapid-air test.

6. All the specimens with entrained air regardless of aggregate (series II) and specimens with the most durable aggregate, even without entrained air (concrete I A) showed no deterioration up to over 900 cycles in the slow-air method.

7. In all combinations of concrete and test method in which there was a difference between the three aggregates, the order of durability was as follows: A most durable, B intermediate, and C least durable.

8. The slow-water method was in general the most severe as to the number of cycles required for failure but the rapid-water was most severe as to the length of time of the tests.

9. The slow-air method was least severe.

Acknowledgment:

The non-automatic portions of the system were designed by R. C. Valore, Jr., and built under the supervision of W. W. Middleton. Thanks are also expressed to A. Van Valkenburg, Jr., for the petrographic examination of the aggregates and to W. J. Youden for help with the statistical treatment of the data.

APPENDIX

DESCRIPTION OF FREEZING-AND-THAWING EQUIPMENT

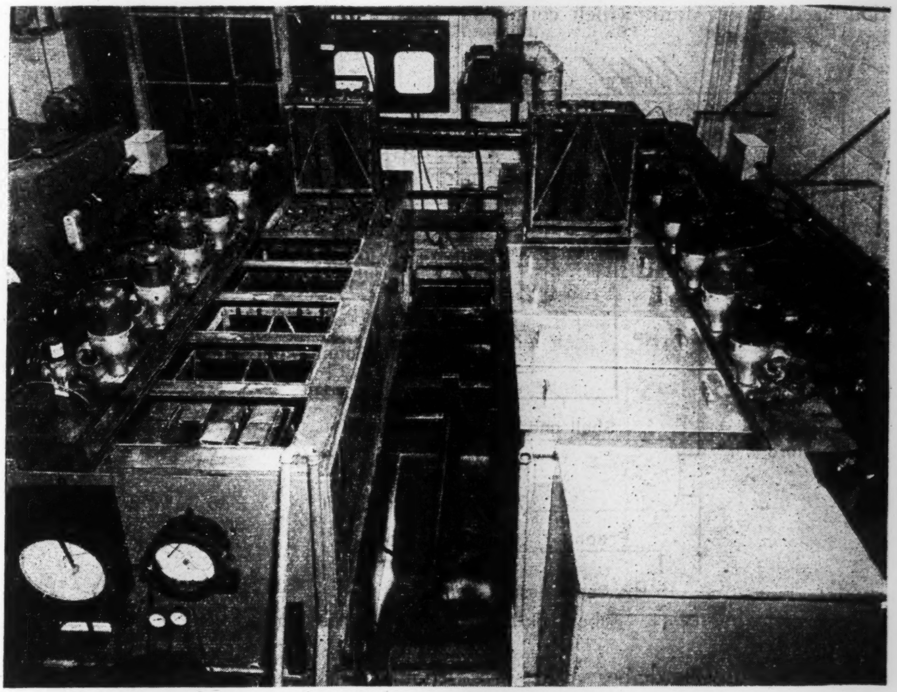

Fig. 10.—Photograph of General Arrangement of Tanks.

Main Assembly:

Figure 10 is a photograph showing the general arrangement of the tanks. Located on each side of the upper level are five 35-gal and one 110-gal specimen treatment tanks. The larger tanks are a part of the rapid-water system which is described in more detail below. On the lower level are located the 300-gal main reservoir and two 110-gal reservoirs. The latter two hold the freezing and the thawing liquid for the rapid-water system.

The tanks are of 18-8 stainless steel, metal-sprayed on the interior with copper and painted with black chemical-resistant laboratory paint. They are insulated from the outside and in most cases from each other by 6 in. of foam glass. Because of its nonabsorptive properties the foam glass insulation makes a hermetically sealed enclosure unnecessary. The stainless steel tanks were coated because in operation it was found that numerous pinholes developed in the bottom of the tanks. This trouble was attributed to local electrolytic action between the stainless steel of the tanks and particles of rust lying on the bottom (7).

A solution of approximately equal parts of ethyl alcohol and water is used as the circulating and cooling medium. Ethylene glycol was originally employed, but proved to have too high a viscosity at low temperature for the pumping system. The use of alcohol introduces some hazard of fire or explosion. As a precaution, the laboratory is maintained at 70 F, which is below the flash point of the solution used. Every tank which contains both alcohol and an electric heater also contains a safety thermostat which cuts off all power to the entire system if the alcohol temperature rises to 70 F.

In use, alcohol is lost through evaporation and by dilution from condensation of moisture from the air. Using 95 per cent alcohol in making up the loss helps maintain the desired proportion of alcohol, but occasionally some of the system liquid must be removed and replaced by 95 per cent alcohol.

Lower Level, Main Assembly:

Figure 11 is a diagram showing the lower-level tanks and also the refrigeration equipment, the latter being located on the floor below. Tank *M* is the 300-gal main reservoir, which is divided into two areas by a loose fitting baffle. A 1-hp pump continuously withdraws liquid from outside the baffle, sends it through the two shell-and-tube coolers in series, then through a closed 275-gal tank and back to the main reservoir, where it is discharged through a manifold along the top of the area inside the baffle. The pipe is 2-in. copper tubing and the rate of flow is approximately 60 gal per min. Each shell-and-tube cooler is flooded with liquid Freon 12 from a 15-hp condenser. Piping and coolers are insulated with 4 in. of cork. The 275-gal tank serves to give temperature stability to the main reservoir by reducing the temperature rise in it when approximately 100 gal of 0 F liquid is returned each hour from the rapid-water system.

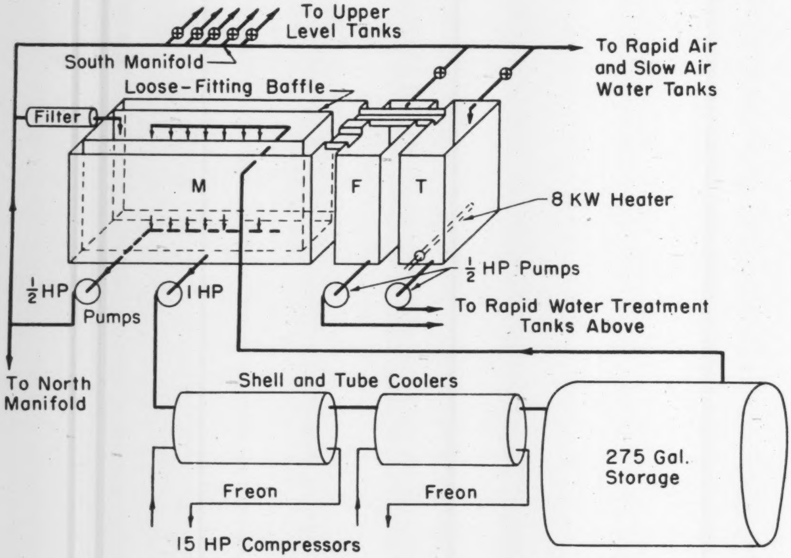
To Upper Level Tanks
South Manifold
To Rapid Air and Slow Air Water Tanks
Loose-Fitting Baffle
Filter
M
F
T
8 KW Heater
½ HP Pumps
½ HP
1 HP
Pumps
To Rapid Water Treatment Tanks Above
To North Manifold
Shell and Tube Coolers
275 Gal. Storage
Freon
Freon
15 HP Compressors

FIG. 11.—Schematic Diagram of Reservoirs and Coolers.

The main reservoir has at various times been maintained at temperatures ranging from −40 F to +12 F. It is currently maintained at approximately −12 F, a temperature which permits more efficient operation of the refrigeration system and the use of less alcohol than is required for lower temperatures. Data reported in this paper, however, were obtained with tank *M* at −20 F. A gas-actuated thermostat with a differential of 3 F controls the temperature by turning the compressors on and off. Two of these thermostats are connected in series with one set a little below the other to prevent freezing in the chillers in case one should fail to act. An automatic recorder graphs the temperature of the reservoir. The suction pressures of the two compressors are recorded on a two-pen recorder with a range of 30 in. of mercury vacuum to 100 lb pressure. These three charts and two common pressure gages for the high sides of the compressors have been of much value in recognizing and diagnosing troubles in the cooling system.

A second pump, of ½-hp capacity, continually takes liquid from a manifold inside the baffle in the bottom of the reservoir and distributes it as needed to various parts of the equipment. The distribution system includes a manifold which runs the length of each bank of treatment tanks (Figs. 10 and 11). Liquid enters the individual tanks through solenoid valves, and excess liquid overflows by gravity into the main reservoir, entering outside the baffle. Both manifolds are bled back to the reservoir through a ceramic filter. Other lines from the pump lead, through solenoid valves, to the freezing and the thawing reservoirs of the rapid-water system (tanks *F* and *T* of Fig. 11), and the thawing-water tanks of both the rapid-air and the slow-air systems. The latter two tanks, which contain water at 40 F, are in equipment adjacent to the main assembly (see below). In them the alcohol circulates through coils of copper tubing and is then piped back to the main reservoir.

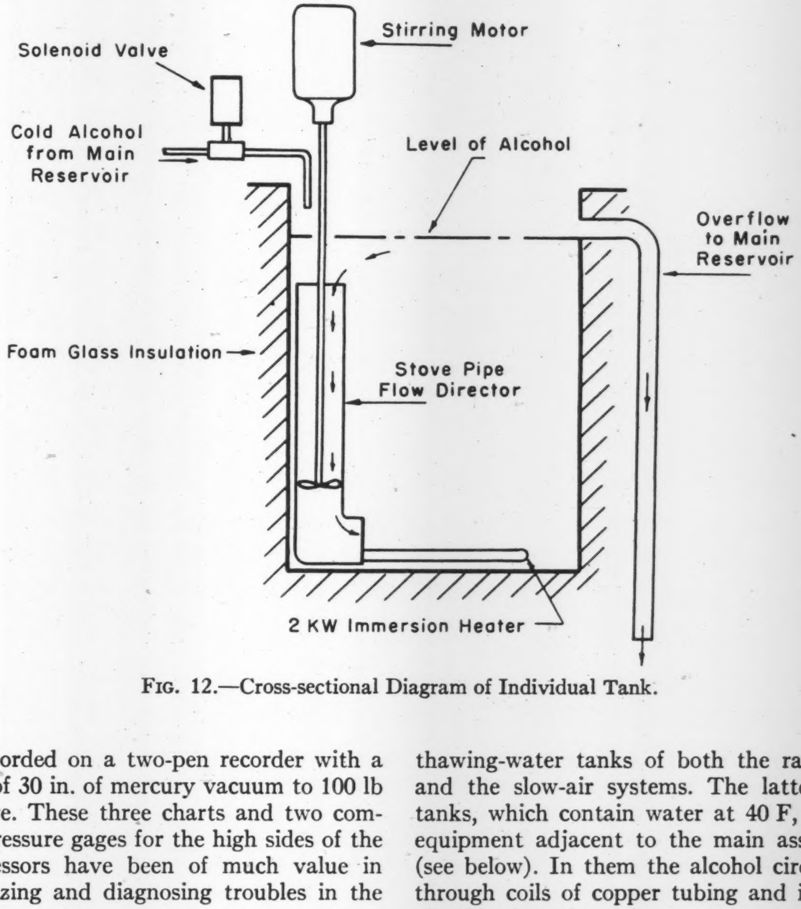

FIG. 12.—Cross-sectional Diagram of Individual Tank.

Tank *F* holds the 0 F alcohol which is required in the freezing phase of the rapid-water system. The temperature is thermostatically controlled by an indicating controller with an adjustable range from −40 to 120 F which opens a solenoid valve, as needed, to add cold alcohol from the main reservoir. Excess alcohol is carried by an overflow trough back to the reservoir.

Tank *T* holds the 42 F alcohol which is required in the thawing phase of the rapid-water system. The temperature is controlled by another indicating controller equipped with a single-pole double-throw mercury switch with an adjustable neutral range. On temperature rise the controller actuates a solenoid valve which permits cold alcohol to enter the tank from the manifold described above, and on temperature fall it energizes two 4-kw immersion heaters. It has been found that the heat loss to the adjacent 0 F tank and to the frozen specimens and their surrounding layer of ice during thawing makes the addition of cold alcohol unnecessary. As a result make-up alcohol must occasionally be added.

Upper Level, Main Assembly:

Figure 12 shows a cross-sectional view of one of the 35-gal treatment tanks. Circulation is provided by a stirrer driven at 1725 rpm by a $\frac{1}{8}$-hp motor. The temperature of the tank liquid is indicated and controlled by an indicating controller in the same manner as the thawing reservoir described above. The temperature of the tank may be varied and controlled at any point between the temperature of the liquid in the main reservoir and 120 F. No tank containing alcohol, however, is ever heated above 70 F. Each tank will accommodate eight 3 by 4 by 16-in. specimens which are enclosed either in cans or rubber boots. The specimens are carried in a rack so that all may be withdrawn at one time by means of an electric hoist on an overhead track.

Adaptation to Slow-Water Cycle:

In order to meet the requirements of ASTM Method C 292[4] it is necessary to control the rate of both heating and cooling. Also the top temperature of this cycle, 73 F, is close to the critical temperature for an evaporation and explosion hazard for an alcohol bath. Six of the 35-gal tanks were modified to meet the requirements and objections given above. First the tanks were isolated from the main system by (1) stopping the overflows and (2) sending the cooling alcohol through loops of $\frac{3}{8}$-in. outside diameter copper tubing in the bottom of the tanks and thence back to the main reservoir. The tanks were then filled with ethylene glycol solution, about 40 per cent by volume. The rate of cooling in each tank is controlled by a needle valve in the alcohol line and is adjusted by timing the rate of flow from a pet cock located just past the solenoid valve. The heating rate is adjusted by a Variac which regulates the voltage to a 500-w heater. A timer operates a stepping relay which alternately shifts the controls from heating to cooling and back again at 24-hr intervals.

The end temperature on the thawing phase of the cycle, 73 F, is limited by a thermostat. Since the heat is cut off very nearly at room temperature, the temperature in the tank stays practically constant without any further heating or cooling until the start of the freezing phase.

The end temperature of the freezing phase, 0 F, is limited and held by the indicating controller mentioned above. Aside from daily observation of the temperature and occasional adjustment of the rate of alcohol flow, the equipment operates without attention seven days a week.

Rapid-Water System:

The main components of the rapid-water system are two treatment tanks on the upper level (Fig. 10), the two reservoirs on the lower level (tanks *F* and *T*, Fig. 11), two $\frac{1}{2}$-hp circulating pumps, two valve motors operating four plug valves, and an automatic timer. The reservoir tanks have been described above.

Each of the treatment tanks holds three racks of eight specimens with the specimens held upright in copper specimen cans or rubber bags. The cooling or heating medium, which is the 0 F or 42 F alcohol from one of the lower reservoirs, enters the bottom of the tank and is distributed by a spray manifold of copper pipe. The manifold runs the length of the tank at the back and has an arm extending under each of the three specimen racks. This system promotes circulation and uniform temperature distribution throughout the tanks and around all the specimen cans.

Each tank has an overflow which can be shifted to empty into either of the lower reservoirs, tank *F* or tank *T*. Also, each tank has two drains which are closed by closet-tank-type rubber balls and are operated by solenoids located above the tanks. One drain from each tank empties into tank *F* and the other into tank *T*.

Figure 13 is a diagram of the operation of the system when the south treatment tank is freezing and the north tank is thawing. The heavy lines indicate the pipes used both on freezing and thawing phases, and the heavy dotted lines (except the drains which are closed during the pumping period) show lines that are being used only for the current phase. At the end of this period, the pumps stop and the appropriate drains are opened. After the draining period, the valve motors shift the plug valves, and automobile choke cables attached to one valve motor shift the hinged overflows to the opposite reservoirs. The liquid is then pumped through the pipes indicated by the light and heavy solid lines in the diagram, overflowing into the proper reservoirs. This cycle repeats continuously.

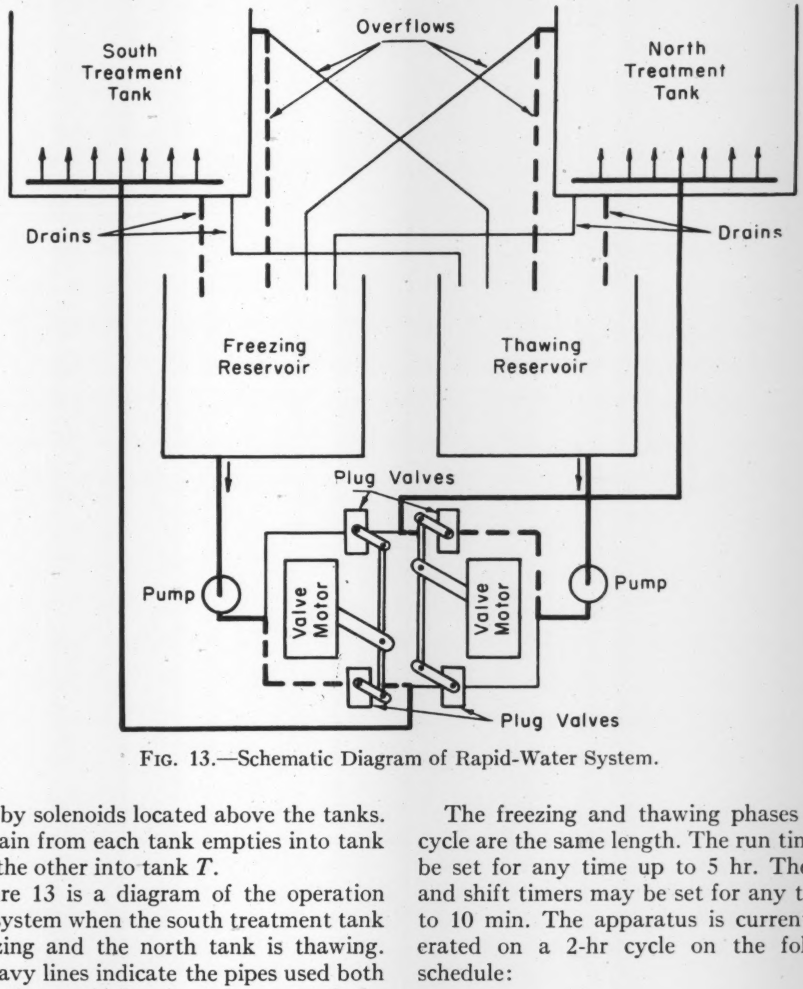

Fig. 13.—Schematic Diagram of Rapid-Water System.

The freezing and thawing phases of the cycle are the same length. The run time may be set for any time up to 5 hr. The drain and shift timers may be set for any time up to 10 min. The apparatus is currently operated on a 2-hr cycle on the following schedule:

Run	56 min
Drain	3 min 50 sec
Shift	10 sec

It was found that specimens could be

stored frozen in this system by merely cutting off the thawing pump and allowing the freezing pump to run every other hour. The insulation of the tanks and the layer of ice in the cans around the specimens keep the temperature in the specimens from rising more than the 3 F above zero allowed by the method[3] during the idle hour. Either treatment tank may be run independently of the other or allowed to run while the other remains frozen. Also, an auxiliary 24-hr timer can be set so that any desired number of thawing cycles may be skipped by either tank (while the other continues freezing and thawing) or by both tanks. This is used to cut down the number of cycles to 30 over a week end or to a smaller number of cycles during other periods when the equipment is unattended, particularly when specimens are just starting in the test or are close to failure.

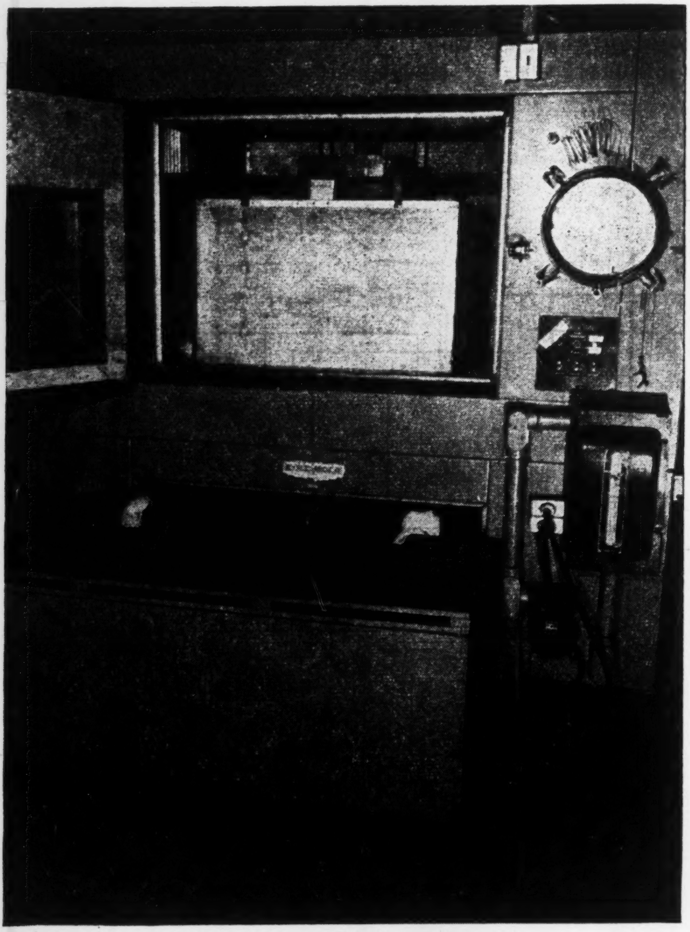

FIG. 14.—Photograph of Rapid-Air Box.

Temperature in this system is controlled, as mentioned above, by two indicating controllers with bulbs in the hot and cold reservoirs.

In order to obtain uniform temperature conditions in the specimens, it was found necessary to have the same amount of water around each one in the specimen cans. The cans were deformed by either pounding the sides in or spreading them until 700 ml of water just reached the top of a new specimen. The specimens are placed in the cans with 3 by 4 by $\frac{3}{4}$-in. paraffin-impregnated wood spacers at top and bottom, the top spacer being weighted down. This provides uniform temperature treatment throughout the length of the specimen.

Strips of rubber refrigerator-door gasket cemented around the top of the can just below the edge of the lid help to keep the spacing uniform between the cans so that the alcohol can circulate freely. These strips also prevent the alcohol from being sprayed up under the lids and into the cans at the start of the pumping period.

The specimen cans in one of the racks may be seen in Fig. 10.

Rapid-Air System:

The rapid-air tests are conducted in a commercial cold box (Fig. 14) which has been modified to operate similarly to the National Sand and Gravel Association apparatus (**4**). This is a low-temperature box with a door opening on the front. Cooling coils are located in chambers at each end of the box, and two blowers draw air through the coils and blow it out into the working chamber through baffles near the top of the end walls. The box has its own refrigeration system consisting of two stages. The first or high stage is 10 hp; the second or low stage is 15 hp and cuts in at a temperature controlled by a separate thermostat. The refrigerant is Freon 22. A recording-indicating controller with a range of -100 to $+200$ C controls the temperature in the box by cutting the compressors off and on after the control setting is reached. The rate of freezing may be controlled either by closing down a valve in the refrigerant return line or adjusting the temperature at which the low-stage machine cuts in. In practice the system has been run with the valve wide open and the low-stage thermostat set at 20 F.

To adapt this box for the rapid-air tests, a galvanized iron tank was installed inside the freezing chamber. In the bottom of the tank a rack of $\frac{3}{8}$-in. pipes spaced approximately $\frac{1}{2}$-in. apart supports the specimens above the bottom of the tank. This allows the freezing air and thawing water to circulate around and under the specimens.

A 16-in. fan in a short cylindrical metal duct is suspended from the roof of the box over the center of the tank. A removable hinged sheet-metal cover is supported 4 in. over the tank on shelves around the back and ends of the freezing box and fits against the duct around the fan. Thus the cold air from the blowers is directed into the central fan, which blows it down into the center of the tank, under and between the specimens, and out over the sides of the tank underneath the lid. The fan is mounted on a shaft which extends through the top of the freezing box and is belt-driven by a 1-hp motor. During the tests reported in this paper, the fan was run at 2300 rpm.

The tank was originally designed to hold 48 3- by 4- by 16-in. specimens. However, extensive tests with different arrangements and different methods of distributing the air showed that it was impracticable to obtain uniform temperature distribution among this number of specimens. Uniformity was finally obtained by reducing the number of specimens to 24 and standing them in a single ring with the air from the fan directed down through the center of the ring.

The thawing water for the system is contained in an insulated tank located in front of the freezing box (Fig. 14). Cooling of the water is accomplished by a cooling coil of $\frac{3}{8}$-in. outside diameter copper tubing through which circulates cold alcohol from the main system described above. Heating is furnished by two 4-kw immersion heaters. An indicating controller opens a solenoid valve in the cooling line or turns on the heaters, as needed. A compressed-air manifold in the bottom of the tank under the cooling coil bubbles air through the water and provides sufficient circulation to keep the temperature uniform when the water is not being pumped and to keep the water from freezing on the coil. Water from the reservoir is pumped up by a 1-hp pump into the bottom of the specimen tank where it is spread by a baffle, circulates around the specimens, and overflows back into the reservoir from the top of the specimen tank through a 3-in. overflow. Pumping provides sufficient circulation for uniform temperature distribution. A 1½-in. drain line controlled by a solenoid valve bypasses the pump so that the water is returned quickly to the reservoir at the end of the thawing phase.

A simple automatic timer and stepping relay control the system automatically. The freezing system, blowers, and fan run for 1 hr. Then there is a 15-min idle period, during which the specimens remain within the specified limits of the freezing temperature. The pump then runs for 30 min, and 15 min are allowed for draining, after which the cycle repeats.

This system may be made to skip cycles and hold the specimens in a frozen condition by removing some of the pins from the timer dial. If the specimens are stored for more than a day or two, however, they are first removed and sealed in two layers of sheet plastic to prevent drying.

Since the freezing coils are not immersed in the thawing water, they gradually frost up during operation and the space between the fins may become so restricted as to materially cut down the air flow and the efficiency of refrigeration. To obviate this, regular defrosting is necessary. It has been found adequate to defrost each time the specimens are removed for sonic tests (three times per week minimum). At the beginning of the last thawing period before the specimens are to be tested, the door is opened and the blowers (but not the fan) are run, with the refrigerators off. This is sufficient to keep the frost from building up.

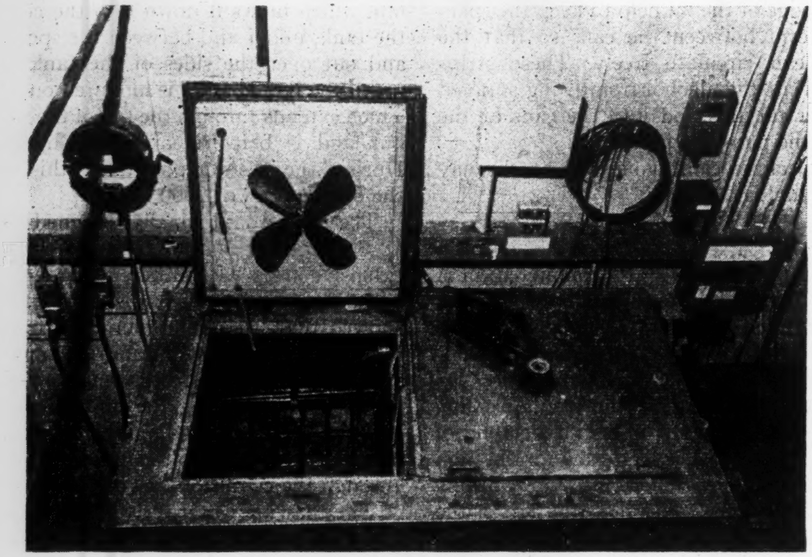

FIG. 15.—Photograph of Slow-Air Box.

Slow-Air System:

The apparatus for the slow-air system is another commercial box of lower freezing capacity than the rapid-air box (Fig. 15). The box has two identical sections with top-opening lids. Cooling coils are mounted in a helical arrangement around the inside of each section and are fed, in parallel, through separate expansion valves from the same compressor and air-cooled condenser. The compressor is a 1-ton unit using Freon 12 which is run by a 2-hp motor. The two chambers are lined with copper and are watertight, which made it unnecessary to install a separate specimen tank.

A rack of ⅜-in. pipe supported on a wooden frame, similar to that in the tank of the rapid-air system, supports the specimens with their lower ends level with the bottom of the cooling coils. Each section holds 24 3- by 4- by 16-in. specimens standing on end. In the lid of each section is a fan which drives the air down among the specimens to the bottom of the box, where it spreads out and rises over the cooling coils around the sides of the box. Due to the comparatively slow rate of cooling, little difficulty is experienced in obtaining a uniform rate of cooling, even between specimens in the center of the box and on the outside near the cooling coils. Needle valves in the refrigerant return lines and adjustable expansion valves permit regulation of the freezing rate of either chamber. The minimum temperature during freezing is set by a pair of refrigeration-type thermostats with a differential of 3 F, one controlling the solenoid in the liquid-refrigerant line for each of the chambers.

For thawing water an insulated tank was installed underneath the freezing box. A $\frac{1}{3}$-hp pump delivers water from the tank into the bottom of each of the chambers. The water flows between the chambers through a 3-in. overflow in the partition and overflows from the left chamber back into the reservoir below. Baffles over the intakes in each box distribute the flow, thus providing uniform temperature distribution. Thawing water temperature is maintained and the treatment tanks are drained in the same manner as in the rapid-air equipment.

The system is controlled automatically by a timer which turns on the fans and opens the refrigerant solenoids (through their temperature controls) at the beginning of the freezing period. At the end of the freezing period the fans are stopped, the cooling solenoids are closed, and the pump starts.

Temperature Measurements:

Temperature changes in all of the systems are followed by copper-constantan thermocouples embedded in the center of dummy specimens. The thermocouples are inserted in $\frac{1}{8}$-in. outside diameter plastic tubing with the junction approximately $\frac{1}{4}$-in. in from the end of the tubing. The tubing is then filled with paraffin. Temperature readings are made with an automatic 16-point recording strip chart potentiometer with a range of −25 F to 125 F graduated to 0.5 F. Terminal strips connected to the potentiometer are located in boxes convenient to all of the freezing tanks and chambers. Leads on the dummy specimens are long enough so that connections may be made from any part of any of the systems to any of the 16 points on the potentiometer.

Sonic Equipment

Deterioration of the specimens during freezing and thawing is checked by measuring the dynamic modulus of elasticity as described in ASTM Method C 215.[9] The specimens are all tested in transverse vibration on a sponge rubber pad. The indicating device is an oscilloscope.

The oscillator is an interpolation type with a dial graduated to 5 cycles in the lower range and 20 cycles in the upper range used in these tests. During this program the oscillator frequency was checked against a steel reference bar for which a number of transverse, torsional, and longitudinal resonant frequencies were known. Variation of the oscillator frequency has always been well within the ±2 per cent specified over the entire range in use, and has almost always been within ±1 per cent.[12]

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[12] Since these tests were completed, an electronic counter has been acquired. This is connected to the oscillator output and counts and displays the frequency directly to the nearest cycle. This eliminates the need for calibrating the oscillator dial.

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DISCUSSION

MR. T. F. WILLIS[1] (*by letter*).—For as long as this writer has dealt with concrete technology, almost every experiment with which he is familiar has occasioned considerable difference of opinion as to the proper interpretation to be placed on the data obtained. Six years of experience with use of statistical methods for design of experiments and analysis of data have produced convincing evidence that more widespread understanding and use of these techniques will go far toward clearing up this condition. It is therefore encouraging to encounter an increasing number of papers, in which the inferences and conclusions are based on statistical analyses of the data. The authors of the paper under discussion are to be commended for being among the early few who have published such a paper.

In the early attempts to use statistical techniques in any branch of technology, many faulty and illegitimate applications are certain to occur. Conclusions, based on such misapplication, can be misleading, or even completely erroneous. The sooner these errors can be detected and pointed out, the quicker will statistics become a universally useful tool for investigations of concrete. While the authors of the paper under discussion do not provide all the information necessary to check carefully the applicability of the statistical techniques they used, superficial examination indicates that most of their applications are correct, and that the results of these applications have enhanced the value of the experiment and paper. At one point, however, there appears to be an improper use of a statistical technique.

Under the rubric "Separation of Specimens within a Given Concrete," the authors examine certain sets of their data by separating the data of a set into two subsets or groups. One group consists of all the larger test results of a set, while the other group contains all the smaller results in the set. A statistical "*t*" test is then applied and, because of the magnitude of the arithmetical result, it is concluded that there is a significant difference between the means of the two groups. This appears to be an illegitimate application of a statistical "*t*" test for the purpose of demonstrating the existence of a real difference between two means. The mechanics of a "*t*" test, and the logic behind inferences drawn therefrom, are based on a theorem which may be stated as follows: given a set of magnitudes normally distributed about the mean of the set, the means of random samples drawn from the set will be distributed in a "*t*" distribution. The connotation of "random," as used in the theorem to restrict "samples," is simply that each individual member of the original set must have had an equal chance of falling in any specific sample. In the authors' application there were two samples or groups, one containing the larger results and the other the smaller. Under their system of forming groups there was no chance for one of the larger results to have occurred in the group of smaller results, nor for one of the smaller results to have fallen in the group of larger results; therefore the authors' groups are in no sense random samples. It follows that there is no *a priori* reason to expect the means of such samples to follow a "t" distribution, which removes the logical background for inferences. The arithmetical result of the "t" test thus appears to be meaningless.

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TABLE IX.

Concrete II B			Concrete II C		
Individual D.F.[a]	Differences	Average Difference	Individual D.F.[a]	Differences	Average Difference
RAPID-WATER METHOD					
78.2 76.5 75.6 69.9	1.7 0.9 5.7	2.8	37.2 33.8 30.4	3.4 3.4	3.4
	13.9			6.8	
56.0 52.2 45.4	3.8 6.8	5.3	23.6 23.6 23.4 23.4 22.8	0 0.2 0 0.6	0.2
RAPID-AIR METHOD					
103.0 102.1 100.6 100.4 99.8	0.9 1.5 0.2 0.6	0.8	97.5 96.5	1.0	1.0
	5.3			6.1	
94.5 94.2 91.3	0.3 2.9	1.6	90.4 86.4 85.3 82.4	4.0 1.1 2.9	2.7
				11.9	
			71.3 62.4	8.9	8.9

[a] Durability factor.

Upon reflection it is obvious that, if any set of test results can be and is separated into two subsets, one containing all of the smaller results and the other all the larger results, then the two subset means will invariably differ from each other in the same algebraic direction. No statistical test is required to substantiate this; it could have been unerringly predicted before performing the experiment. Furthermore, the fact that it is so reveals nothing about any other characteristic of one subset relative to the other.

The authors hypothesize that the occurrence of both small and large results, within a single set of tests from supposedly identical specimens, is due to the presence or absence of non-durable aggregate particles at critical positions in a specimen. This may or may not be true; but for reasons pointed out above, the arithmetical result of a "t" test, applied under the authors' method of assigning specimens to groups, lends no authenticity to this or any other similar hypothesis. Had the authors, prior to acquiring knowledge about the magnitude of the frost resistance of the individual specimens, been able to separate them into two groups on the basis of observed location of non-durable aggregate particles, the result of a "t" test on the difference between the mean frost resistances of the two groups would have been meaningful.

A fallacy, such as has been pointed out, can be avoided by remembering an inviolable principle of scientific experimentation, to wit: if groups of experimental units are to be compared for the purpose of demonstrating a difference (or similarity) between such groups, placement of units in the groups must in no way be influenced by the measured magnitudes which are to serve as the basis for comparison.

In closing, the writer would emphasize

that the enhanced value of the paper, resulting from the legitimate uses of statistics, far outweighs the erroneous inference on a minor point produced by misapplication of a statistical technique; and further would urge the authors and all technologists concerned with applied research on concrete to continue and expand their usage of statistical techniques for design of experiments and analysis of data.

MR. H. T. ARNI (*author's closure*).—We wish to thank Mr. Willis for his careful and critical review of our paper, and for the discussion of the statistical procedures used therein. Mr. Willis' criticism of the use of the "*t*" test to demonstrate a significant difference between the averages of groups of data is well taken and the use of this statistical technique was improper. It should have been pointed out that the actual separation between these groups of data was clearly obvious without any statistical analysis in the original curves where the relative durabilities of the individual specimens were plotted. These separations appeared early in the life of the specimens and continued to the end of the tests, with the gaps between the groups growing progressively larger. Table IX shows the gaps that existed between groups by showing the differences between the durability factors of successive specimens (in decreasing order of durability) together with the average differences for the groups.

The hypothesis of non-durable aggregate particles in critical locations was presented as a possible explanation for the appearance of such clearly separated groups in specimens from the same concrete. The only evidence supporting such a hypothesis is that the groups appeared only in the two concretes that contained occasional pieces of non-durable aggregate that were known to be responsible in some cases for failure of otherwise sound specimens.

This point was presented merely as an interesting piece of evidence suggesting that the two rapid freezing-and-thawing methods had brought out differences in the specimens that were not originally known to be there, a result which would be desirable in any method which was to be used seriously as a test method.

CORRELATION OF SONIC PROPERTIES OF CONCRETE WITH CREEP AND RELAXATION*

By Tien S. Chang[1] and Clyde E. Kesler[1]

Synopsis

This paper presents a method of relating the properties of concrete that can be obtained from sonic testing to the creep behavior in compression and flexure and relaxation behavior in compression. Valid non-linear mechanical models are selected to represent concrete in creep and relaxation and their coefficients are statistically related to the sonic properties. A simple procedure is also developed to relate the creep of one concrete beam to another under different loading.

The results presented provide a means of predicting creep and relaxation behavior of concrete from a relatively simple sonic test.

The purpose of the investigation discussed here was to determine if a correlation existed between the creep and relaxation behaviors and the sonic properties of concrete. The theory of rheological models was used as the means of obtaining a correlation.

Reiner (**1**)[2], Freudenthal (**2**) and others in studies they have made have considered concrete to be a viscoelastic material. This concept has not had wide acceptance. Nevertheless, if it is correct, a correlation should exist between the dynamic behavior and static behavior of concrete. The authors have previously reported successful use of this concept in studies of creep of concrete (**3**).

Scope of Investigation

Two of the simplest rheological behaviors of any viscoelastic material are the creep strain-time relationship under a constant load and the relaxation stress-time relationship under a constant deformation. Both of these relationships were considered together with properties obtained from sonic-testing of concrete.

The investigation included five series of tests as follows:

1. Short-time (up to three days) creep in compression.
2. Long-time (up to seven months) creep in compression.
3. Relaxation in compression.
4. Creep of uniformly loaded beams.
5. Creep of beams with concentrated loads.

The cylinders used in the creep and relaxation studies were tested in a resonant type sonic apparatus immediately before being subjected to the static tests. In the case of beams, the sonic properties were obtained from companion cylinders.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] Instructor and Associate Professor of Theoretical and Applied Mechanics, respectively, Univ. of Illinois, Urbana, Ill.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1271.

SPECIMENS

All specimens were made with type I portland cement and natural aggregates. The aggregates are from the Wabash River near Covington, Ind., and are part of a glacial outwash probably from the Wisconsin glaciation. Sieve analyses of the aggregates are given in Table I. The absorption of the aggregates was about one per cent by weight of the surface dry aggregates. Both of these aggregates pass the usual specification tests.

Different mixes were used and the strength at the time of test varied from 1100 to 5880 psi. The slump varied from 1 to 8 in. Specimens were moist cured for different lengths of time and then were stored in a controlled environment at 76 F and 50 per cent relative humidity. Tables II, III, and IV give the slump, length of moist curing, mix and age at time of test for the various specimens.

TABLE I.—SIEVE ANALYSIS OF AGGREGATES.

Sieve	Per Cent Passing	
	Sand	Gravel
1 in.	...	92.6
¾ in.	...	68.9
½ in.	...	53.3
⅜ in.	...	24.9
¼ in.	...	1.7
No. 4	98.3	1.0
No. 8	88.3	...
No. 16	61.7	...
No. 30	39.0	...
No. 50	9.5	...
No. 100	1.8	...

All cylinders were of the standard size, 6 by 12 in. The beams, subjected to uniform load were 4 by 6 by 100 in., the 4-in. dimension being vertical. The beams subjected to concentrated loads were 6 by 6 by 64 in. Companion cylinders were made with each beam. Some of these cylinders were subjected to sonic, creep and relaxation tests.

EQUIPMENT AND TESTING PROCEDURES

Sonic tests:

The sonic apparatus used in this investigation and the manner in which it is used has been described previously (4). The equipment, shown diagrammatically in Fig. 1, is used to determine the resonant modulus of elasticity and the logarithmic decrement—the two sonic properties used in this study.

Important components for these tests are the frequency counter and the vacuum tube voltmeter. Both are necessary in determining sufficiently accurate values of the logarithmic decrement, which is a measure of damping. The voltmeter is necessary to assist in obtaining nearly constant power input for the various frequencies and to locate frequencies that resulted in amplitudes of 0.707 of the maximum, since the logarithmic decrement was determined as follows:

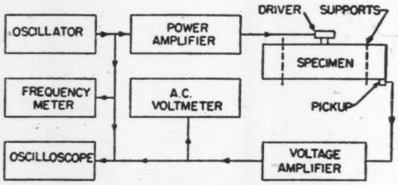

FIG. 1. Schematic Diagram of Sonic Apparatus.

$$\delta = \pi \frac{(f_2 - f_1)}{f_0} \qquad \text{...........(1)}$$

where:

δ = logarithmic decrement,
f_0 = resonant frequency, and
f_1, f_2 = frequencies on either side of resonance at which the amplitude of vibration is 0.707 of the maximum.

Since the term $(f_2 - f_1)$ is small compared to the magnitude of either f_1 or f_2 a frequency counter is necessary to obtain sufficient accuracy.

TABLE II.—EXPERIMENTAL DATA AND RESULTS OF ANALYSIS OF CREEP IN COMPRESSION.

Cylinder Number	Mix	Slump, in.	Curing-Period, days	Age at Test, days	Strength, f_c', psi	Dynamic Modulus, E_d, psi	Logarithmic Decrement, δ	Stress, σ, psi	ϵ_{c_1}, in. per in.	$1 - n\epsilon_c$
A1a1.....	1:3.1:4.5	8	6	22	3 780	4.56×10^6	0.0332	2460	1.015×10^{-4}	0.723
A1a2.....	1:3.1:4.5	8	6	23	3 560	4.36	0.0333	2300	0.872	0.733
A1a3.....	1:3.1:4.5	8	6	28	3 790	4.36	0.0325	3000	1.685	0.692
A1a4.....	1:3.1:4.5	8	6	30	4 150	4.34	0.0286	2650	0.707	0.731
A1b1....	1:3.1:4.5	8	13	36	3 900	4.51	0.0331	2750	1.219	0.740
A1b2....	1:3.1:4.5	8	13	42	3 600	4.68	0.0344	3230	1.780	0.730
A1b3....	1:3.1:4.5	8	13	25	4 180	4.25	0.0254	2690	0.675	0.680
A1b4....	1:3.1:4.5	8	13	32	3 870	4.82	0.0275	3750	2.200	0.625
A1c1.....	1:3.1:4.5	8	20	93	4 210	4.64	0.0314	3180	1.120	0.719
A2a1.....	1:3.1:4.5	1½	6	97	5 630	4.82	0.0208	3890	1.061	0.679
A2a2.....	1:3.1:4.5	1½	6	100	5 800	4.82	0.0185	4060	1.261	0.674
A3b1....	1:3.1:4.5	3½	13	216	4 970	4.69	0.0225	3000	0.553	0.704
A3b2.....	1:3.1:4.5	3½	13	221	4 750	4.60	0.0268	2050	0.264	0.706
A3b4....	1:3.1:4.5	3½	13	280	4 810	4.64	0.0201	1485	0.094	0.713
A3c1.....	1:3.1:4.5	3½	20	53	5 070	5.11	0.0268	4030	1.303	0.645
A3c2.....	1:3.1:4.5	3½	20	58	5 320	4.68	0.0207	3610	0.874	0.669
A3c3.....	1:3.1:4.5	3½	20	69	5 880	5.07	0.0277	4070	1.585	0.697
A4a1.....	1:3.1:4.5	3½	6	234	4 290	4.55	0.0198	2470	1.430	0.740
A4a2.....	1:3.1:4.5	3½	6	237	4 250	4.50	0.0214	2120	0.230	0.722
A4a4.....	1:3.1:4.5	3½	6	247	4 270	4.52	0.0206	2820	0.440	0.683
A4b1.....	1:3.1:4.5	3½	13	261	4 880	4.70	0.0179	2680	0.350	0.733
A4b2....	1:3.1:4.5	3½	13	264	4 880	4.65	0.0178	3110	0.546	0.728
A4b3....	1:3.1:4.5	3½	13	266	4 780	4.80	0.0181	3070	0.450	0.750
A4c3.....	1:3.1:4.5	3½	20	351	4 300	4.65	0.0251	990	0.070	0.788
C1a5.....	1:5.0:12.0	6½	6	63	1 100	2.99	0.0330	955	1.000	0.742
D4a1....	1:5.7:7.7	1	6	84	2 900	4.08	0.0351	1200	0.294	0.699
D4a5....	1:5.7:7.7	1	6	61	2 300	4.06	0.0302	1240	0.381	0.771
D4a6....	1:5.7:7.7	1	6	61	2 600	4.20	0.0302	1240	0.273	0.746
C5a5.....	1:5.7:7.7	1	6	113	1 420	3.02	0.0268	830	0.370	0.796
C5a6.....	1:5.7:7.7	1	6	113	1 360	3.00	0.0218	830	0.310	0.752
D3a4....	1:5.7:7.7	1	6	156	1 480	2.97	0.0246	583	0.112	0.790
D3a5....	1:5.7:7.7	1	6	156	1 340	3.04	0.0253	583	0.134	0.788
F1a5.....	1:3.1:4.5	1	7	21	3 700	5.01	0.0402	1410	0.190	0.752
F1a6.....	1:3.1:4.5	1	7	21	3 700	5.01	0.0402	1410	0.190	0.752
F3a1.....	1:3.1:4.5	1	6	54	4 530	4.89	0.0263	2470	0.325	0.738
F3a2.....	1:3.1:4.5	1	6	55	4 720	5.18	0.0251	2150	0.222	0.670
F3a5.....	1:3.1:4.5	1	6	56	4 900	5.00	0.0277	1240	0.106	0.791
F3a6.....	1:3.1:4.5	1	6	61	5 500	5.09	0.0253	1060	0.076	0.776
F4a1.....	1:3.1:4.5	1	6	50	4 650	4.77	0.0278	2470	0.450	0.662
F4a2.....	1:3.1:4.5	1	6	53	4 320	4.88	0.0226	3530	0.808	0.687
F4a5.....	1:3.1:4.5	1	6	47	3 400	4.62	0.0319	1060	0.138	0.756
F4a6.....	1:3.1:4.5	1	6	52	4 300	4.85	0.0296	1240	0.113	0.754

TABLE III.—EXPERIMENTAL DATA AND RESULTS OF ANALYSIS OF RELAXATION IN COMPRESSION.

Cylinder Number	Mix	Slump, in.	Curing Period, days	Age at Test, days	Strength, f_c', psi	Dyanmic Modulus, E_d, psi	Logarithmic Decrement, σ	Initial Stress σ_0, psi	$\Delta\sigma_{r_1}/\sigma_0$	$n\Delta\sigma_r$
A1c2...........	1:3.1:4.5	8	20	100	3 500	4.64×10^6	0.0310	3190	0.094	0.177
A3a1...........	1:3.1:4.5	3½	6	217	4 000	4.35	0.0205	2970	0.158	0.110
A3c4...........	1:3.1:4.5	3½	20	45	5 000	5.00	0.0275	3750	0.160	0.129
II1ATI[a]........	......	...	...	...	4 200	4.48	0.0220	2300	0.056	0.238

[a] Foreign Cylinder.

TABLE IV.—EXPERIMENTAL DATA AND RESULTS OF ANALYSIS OF CREEP IN FLEXURE.

Beam Number	Dimensions, in.	Mix	Slump, in.	Curing Period, days	Age, days	Strength, f'_c, psi	Computed Flexural Stress, psi	Dynamic Modulus, E_d, psi	Logarithmic Decrement	Δ_{c_1}, in.	$^n\Delta_c$, in.
Uniform Loading											
C1a	4 by 6 by 100	1:5.0:12.0	6½	6	63	1 100	144	3.11×10^6	0.0329	1.10×10^{-4}	0.535
D4a	4 by 6 by 100	1:5.7:7.7	1	6	58	2 600	144	4.13	0.0299	1.80	0.450
D5a	4 by 6 by 100	1:5.7:7.7	1	6	54	2 600	144	4.16	0.0299	2.20	0.450
F1a	4 by 6 by 100	1:3.1:4.5	1	7	23	3 700	144	5.01	0.0407	3.45	0.393
F4a	4 by 6 by 100	1:3.1:4.5	1	6	52	4 160	144	4.90	0.0280	2.81	0.400
Third-Point Loading											
B1a[a]	6 by 6 by 64	1:3.1:4.5	5	6	455	4 040	...	4.56	0.0179	...	...
B1b	6 by 6 by 64	1:3.1:4.5	5	6	455	4 040	140	4.56	0.0179	0.43	0.420
G1a[a]	6 by 6 by 64	1:2.4:3.5	1	6	52	6 100	...	5.69	0.0259	...	...
G1b	6 by 6 by 64	1:2.4:3.5	1	6	52	6 100	172	5.69	0.0259	1.25	0.372

[a] Control beams—not loaded.

The frequency counter was also used in determining the resonant frequency which was *located* by observing the maximum deflection of the voltmeter. The resonant modulus of elasticity in flexure was computed using this frequency and the following formula:

$$E_d = Cwf_0^2 \qquad (2)$$

where:

E_d = resonant modulus of elasticity,
f_0 = resonant frequency,
w = weight of specimen, and
C = a factor which depends upon the shape and size of specimen, the mode of vibration, and Poisson's ratio.

All sonic tests were made on 6 by 12-in. standard cylinders. To insure reproducible results, all cylinders were vibrated in flexure in three planes at angles of approximately 120 deg to each other. This procedure tends to reduce the effect of the inherent heterogeneity of the concrete and lack of symmetry in the shape of the specimen should there be any.

Short-time Creep in Compression:

All cylinders for short-time creep tests, up to three days, were loaded in a 200,000-lb Universal testing machine which incorporated an automatic load-sustaining device. The strains were measured automatically over the center 6 in. of the specimen by equipment involving the use of microformers. The time, observed from a stop watch, was marked and recorded at appropriate intervals on the chart which included a continuous record of the load and creep strains. The chart, thus, at the completion of the test provides sufficient information for plotting of creep-time curves and other analytical studies of creep behavior. For the creep test, the cylinders were loaded at the rate of 25 psi per sec to the desired stress.

Long-time Creep in Compression:

Cylinders used for long-time creep tests were all loaded in specially designed frames as shown in Fig. 2. Each frame was capable of loading two cylinders. A spring was included in the apparatus to prevent rapid relieving of the load as the cylinders deformed. Furthermore, the spring served as a rather accurate means of measuring the load on the specimens.

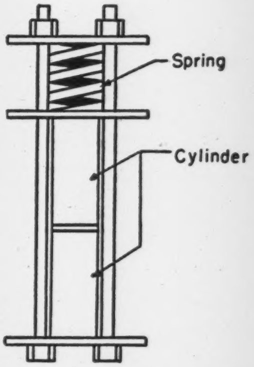

Fig. 2.—Apparatus Used for Long-Time Creep Tests.

For each cylinder there was a control cylinder which had the same historical data as that of the active cylinder, and mechanical gage points for Whittemore gage readings as well as SR-4 electrical strain gages were glued to both the control and active cylinders. The Whittemore gage had a 5-in. gage length and the SR-4 gages were type A9 having a 6-in. gage length. A steel control bar was used for checking the Whittemore gage zero-readings and for obtaining the shrinkage of the control cylinders. Another mild steel plate was used for electrical strain gages for the same purpose. In general, one pair of Whittemore gage points was put on the steel bar, one pair on the control cylinder and four pairs on the active cylinder located in the center 5-in. of the cylinder and equally spaced around the specimen. Two electrical strain gages were glued to the steel control plate, two on the concrete control cylinder, and two on the loaded cylinder in the center of the cylinder diammetrically opposite each other.

After initial readings were taken for the various gage lines and gages, each cylinder to be loaded was paired with another and placed in a creep frame as shown in Fig. 2. The initial load was applied through the spring by an Universal testing machine, and the nuts on the long bolts were tightened. After loading, the cylinders were returned to the controlled environment room and readings taken at appropriate intervals.

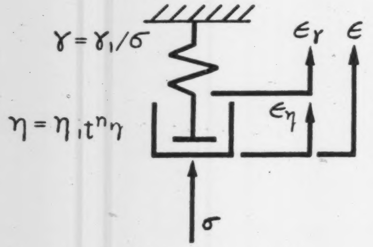

FIG. 3.—Non-linear Maxwell's Liquid.

Relaxation in Compression:

All relaxation tests were made on cylinders in a 200,000-lb Universal testing machine. For each loaded cylinder there was a companion cylinder not under load used to measure the effect of temperature, humidity, and shrinkage. The relaxation tests were made in the ordinary laboratory atmosphere. All strains were measured by electrical resistance strain gages over the center 6-in. of the cylinders.

The cylinder for the relaxation test was loaded in a minimum time of about 30 sec. The strain was noted immediately and then maintained at this level. The load was read at predetermined intervals.

Creep in Flexures under Uniform Loads:

Six beams, 4 by 6 by 100 in., were supported on spans of 96 in. The 4-in. dimension was vertical. The weight of the beams was sufficient to cause creep and no other loads were applied. At the center of the beams, electric resistance strain gages were attached to the top and bottom of the beams and Whittemore gage points were placed on the sides of the beams at four different depths. The deflection of each beam was measured at midspan by 0.001-in. dial gages. For each beam tested, companion cylinders were used for shrinkage measurements and sonic and creep tests.

Creep in Flexure Under Concentrated Loads:

Two beams, 6 by 6 by 64 in., were subjected to third-point loading on a span of 60 in. A constant load was maintained by a lever system and weights. For each beam there was a control beam, control cylinders and several other cylinders of the same concrete which were used for sonic and creep tests. The control beam was supported on a 60-in. span but without an external load being applied.

On both the active and control beams at midspan, seven 6-in. type A9 gages were used, one each on top and bottom and five along the depth of the beam. Center deflections were obtained with 0.001-in. dial gages.

RESULTS AND ANALYSIS OF DATA

Creep in Compression:

Creep of concrete under sustained axial compression can be represented by either of two non-linear rheological models, the Kelvin solid (3) or the Maxwell liquid, Fig. 3. A non-linear Maxwell model will also describe the relaxation behavior and was therefore considered more satisfactory.

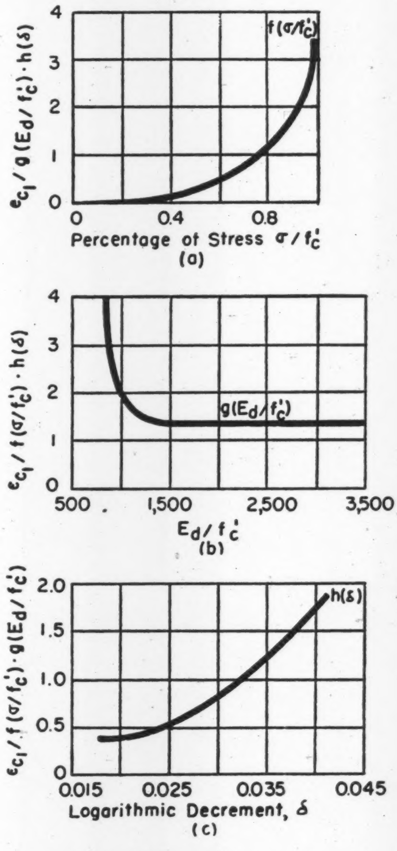

FIG. 4.—Functions of ϵ_{c_1} for Creep in Compression.

The non-linear Maxwell liquid that describes the behavior of concrete is composed of a stress-softening spring and a power-time thickening dashpot as shown in Fig. 3. The equations governing the rheological behavior of the model under relatively small acceleration are:

$$\epsilon = \epsilon_\gamma + \epsilon_\eta$$

$$\sigma = \gamma\epsilon_\gamma = \frac{d\epsilon_\eta}{dt} \quad \text{............(3)}$$

where:

ϵ_γ = strain of the spring,

ϵ_η = strain of the dashpot,

ϵ = strain,

σ = sustained stress,

t = time,

$\gamma = \frac{\gamma_1}{\sigma}$ and is the spring coefficient, and

$\eta = \eta_1 t_c^{n_\eta}$ and is the dashpot coefficient.

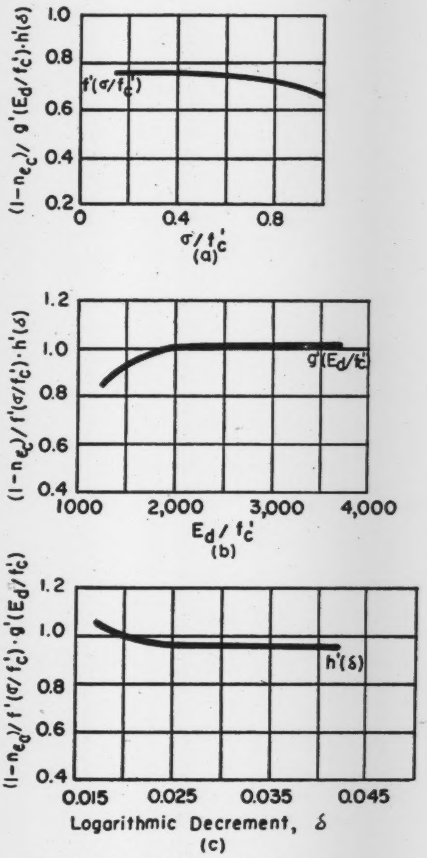

FIG. 5.—Functions of n_{ϵ_c} for Creep in Compression.

By letting σ be constant and solving Eq. 3 the following creep function results:

$$\epsilon_c = \frac{\sigma}{\eta_1(1-n_\eta)}\, t_c^{(1-n_\eta)} \quad \text{.......(4)}$$

where ϵ_c is the creep strain and t_c is the creep time.

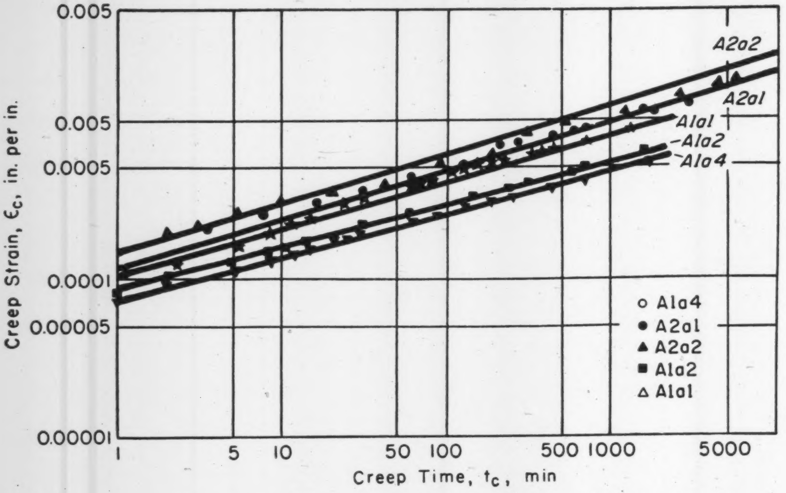

FIG. 6.—Comparison of Theoretical Creep Curve and Experimental Creep Data for Short-Time Tests.

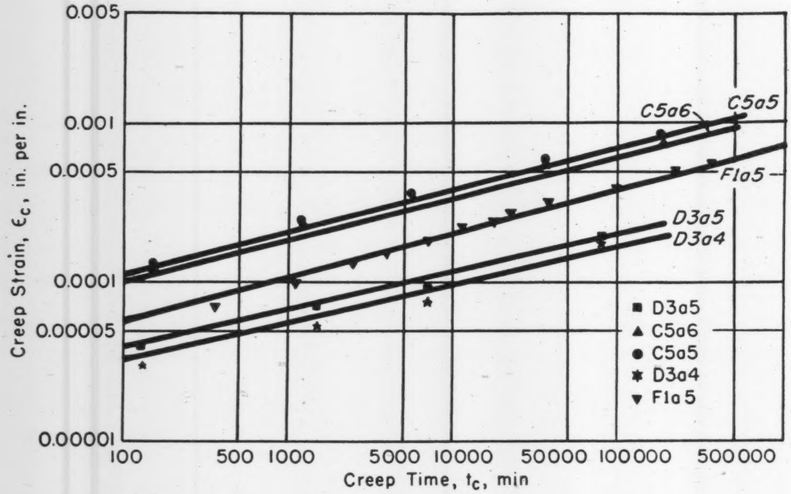

FIG. 7.—Comparison of Theoretical Creep Curves and Experimental Creep Data for Long-Time Tests.

This equation indicates that the creep function of concrete is a power function of time and therefore the creep curves will be straight lines when plotted logarithmically.

From Eq. 4 the following general expression is obtained:

$$\epsilon_c = \epsilon_{c_1} t_c^{n_{\epsilon c}} \qquad (5)$$

where:

$$\epsilon_{c_1} = \frac{\sigma}{\eta_1(1 - n_\eta)}, \quad \text{and}$$

$$\eta_{\epsilon_c} = 1 - n_\eta .$$

Both ϵ_{c_1} and $n_{\epsilon c}$ have been shown, by dimensional analysis and step-by-step approximation, to be functions of the percentage of sustained stress, the ratio of modulus of elasticity to the ultimate strength, and the logarithmic decrement (3) and are as follows:

$$\left.\begin{aligned}\epsilon_{c_1} &= f(\sigma/f'_c) \times g(E_d/f'_c) \times h(\delta)\\ n_{\epsilon_c} &= f'(\sigma/f'_c) \times g'(E_d/f'_c) \times h'(\delta)\end{aligned}\right\} \quad \ldots\ldots\ldots(6)$$

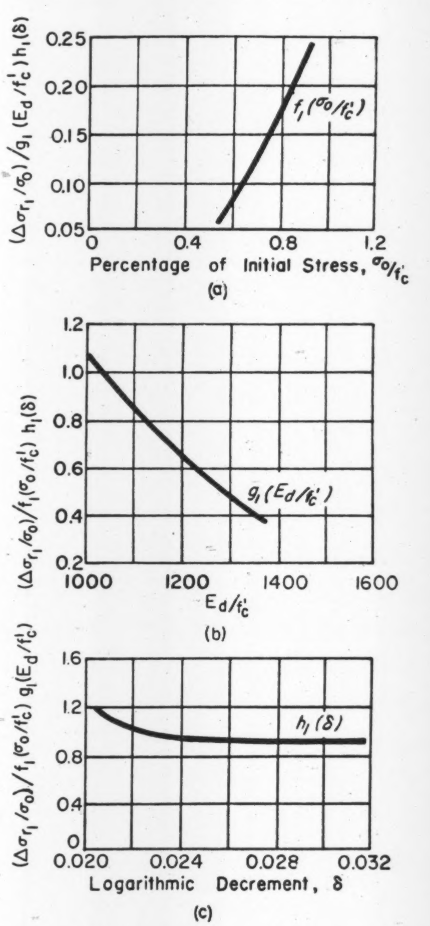

FIG. 8.—Functions of $\Delta\sigma_{r1}/\sigma_0$ for Relaxation in Compession.

These functions are shown in Figs. 4 and 5.

The close agreement obtained between the theoretical curve and experimental data is shown in Figs. 6 and 7.

Relaxation in Compression:

If the same kind of non-linear Maxwell liquid as used in creep is employed to describe the relaxation behavior of concrete under axial compression, the governing equations will be the same as Eq. 3. The solution of these equations for a constant strain and an initial stress of σ_0 is:

$$\Delta\sigma_r = \sigma_0 - \sigma = \frac{\gamma_1}{2\eta_1(1 - n_\eta)}\, t_r^{(1-n_\eta)} \quad \ldots(7)$$

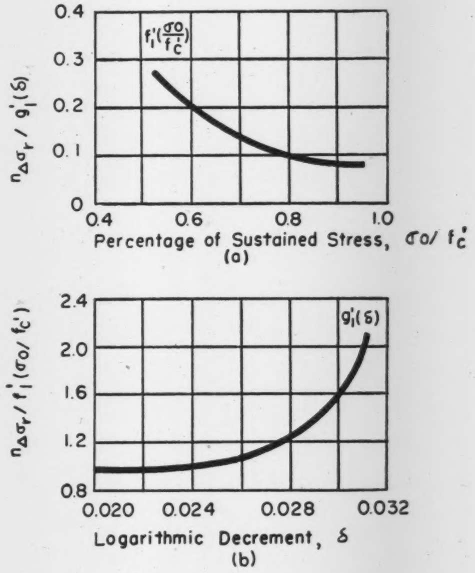

FIG. 9.—Functions of $n_{\Delta\sigma_r}$ for Relaxation in Compression.

where:

t_r is the elapsed time the concrete has been subjected to a constant strain.

If:

$\Delta\sigma_{r_1} = \gamma_1/2\eta_1(1 - n_\eta)$ and $n_{\Delta\sigma_r} = 1 - n_\eta$,

then

$$\Delta\sigma_r = \Delta\sigma_{r_1} \times t_r^{\,n_{\Delta\sigma_r}} \quad \ldots\ldots\ldots(8)$$

The constants $\Delta\sigma_{r_1}$ and $n_{\Delta\sigma_r}$ in Eq 8 were found for the four cylinders tested. The same procedures of dimensional analysis, step-by-step approximations, and grossing of errors were applied to these constants. The results, though not accurate enough so far as statistical analysis is concerned due to the lack of sufficient data, are quite encouraging in pointing out the probable trends of variations of these constants with the sonic properties of concrete cylinders.

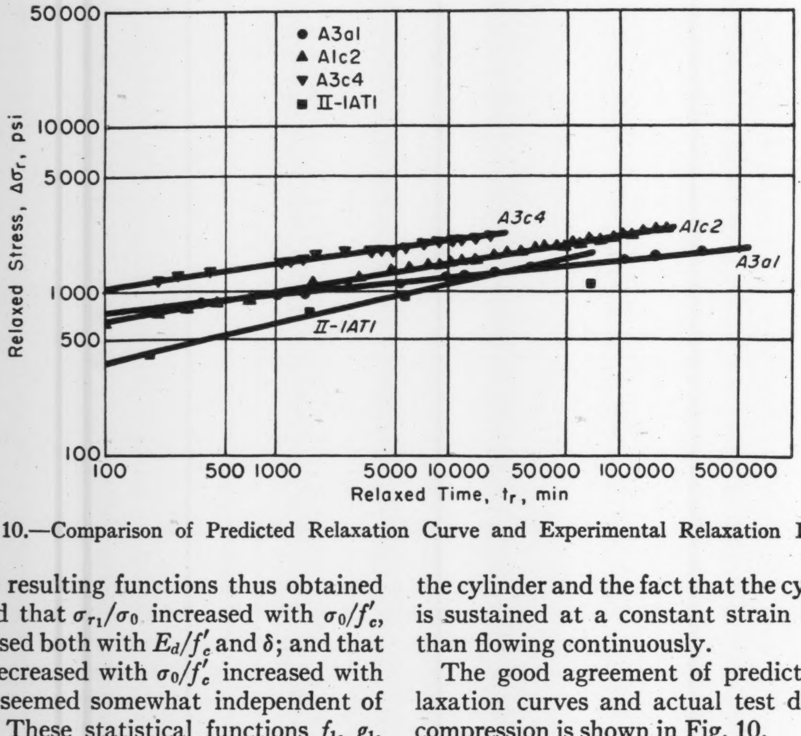

FIG. 10.—Comparison of Predicted Relaxation Curve and Experimental Relaxation Data.

The resulting functions thus obtained showed that σ_{r_1}/σ_0 increased with σ_0/f'_c, decreased both with E_d/f'_c and δ; and that $n_{\Delta\sigma_r}$ decreased with σ_0/f'_c increased with δ and seemed somewhat independent of E_d/f'_c. These statistical functions f_1, g_1, h_1, f'_1 and h'_1 are shown in Figs. 8 and 9.

The values of $\Delta\sigma_{r_1}/\sigma_0$ and $n_{\Delta\sigma_r}$ can be determined from Figs. 8 and 9 and the following equations:

$$\begin{aligned} \sigma_{r_1}/\sigma_0 &= f_1(\sigma_0/f'_c) \times g_1(E_d/f'_c) \times h_1(\delta) \\ n_{\Delta\sigma_r} &= f'_1(\sigma_0/f'_c) \times h'_1(\delta) \end{aligned} \quad \text{..(9)}$$

From the results of the above equations the values of η'_1 and n_η can be found as follows:

$$\eta_1 = \frac{\gamma_1}{2\Delta\sigma_{r_1} n_{\Delta\sigma_r}} \text{..........} (10)$$

$$n_\eta = 1 - n_{\Delta\sigma_r}$$

One important point to be noted is that the exponent of the time function of the thickening dashpot obtained from Eq. 10 is generally larger than that obtained for creep tests. This is probably due to the constant releasing of stress on the cylinder and the fact that the cylinder is sustained at a constant strain rather than flowing continuously.

The good agreement of predicted relaxation curves and actual test data in compression is shown in Fig. 10.

Creep of Uniformly Loaded Beams:

Five 4 by 6 by 100-in. beams were tested in creep under their own weight while simply supported. Since all had approximately the same unit weight, the loading conditions and the magnitudes of loading on these five beams were identical. This eliminated one major variable in the study of creep of concrete under sustained flexure loading. The advantage of this is obvious, but there is also the disadvantage that no conclusions can be made from these test data concerning the correlations between the creep of beams and the loading conditions.

The creep strains[3] in tension and in compression were quite different in magnitude. Creep curves for tension and compression in beam D4a are shown in Fig. 11. This phenomenon of unequal creep of concrete in tension and in compression becomes more evident when the distribution of strain across a beam is considered with time. Furthermore, the logarithmic plots of the mid-span creep deflections of the beams, though still straight lines, showed distinctly greater slopes than those for the cylinders creeping under constant sustained compressive stresses. This is due to the total angle changes along the span of the beam which increase not only with the creep of the concrete but also with the shifting of the neutral axis due to unequal creep of concrete in compression and in tension. It therefore becomes impossible to predict the creep of concrete in flexure directly from the creep data of concrete in axial compression without some empirical adjustment unless there is a thorough knowledge of the creep behavior of concrete in tension. It is obvious that the empirical relationships of these creep data can either be applied to the creep constants that appear in Eq 6 or directly to the sonic properties of their companion cylinders. To apply the analysis to the creep constants, several assumptions must be made, such as the initial stress distribution, the relationship between the creep constants for compression and for tension, and the initial modulus of elasticity in compression and in tension, etc. The limited data available do not indicate which of these assumptions may be justified or erroneous. If it is assumed that the tensile creep constants are also functions of the sonic properties and the ultimate strength, then a direct prediction of the creep curves of concrete beams should be possible from the sonic properties of their companion cylinders without detailed correlations with the creep behaviors of concrete in axial compression.

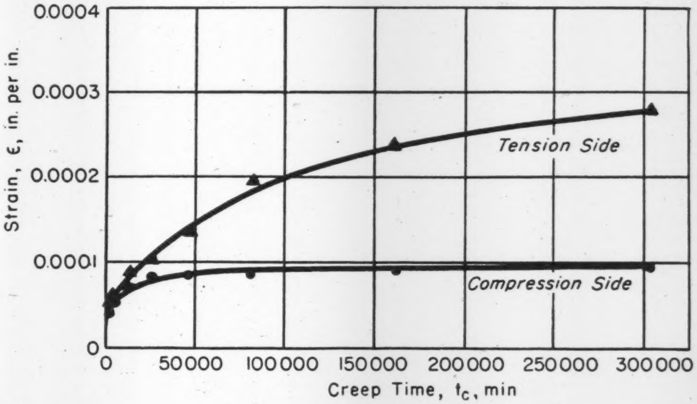

FIG. 11.—Compression and Tension Creep Curves for Uniformly Loaded Beam D4a.

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[3] They are not creep strains in the sense defined earlier, because the stresses are not constant across the depth of the beam with time when the beam is creeping.

It is assumed that the resulting creep deflections of the beams can still be represented by an equivalent model of the non-linear Kelvin or Maxwell type as for axial compression, then the creep curves should also be power functions. This possibility was indicated by logarithmic plots of the mid-span creep deflection and time. Writing this out explicitly, the creep deflection-time relationship is:

$$\Delta_c = \Delta_{c_1} \times t_c^{\,n_{\Delta_c}} \quad \ldots\ldots\ldots (11)$$

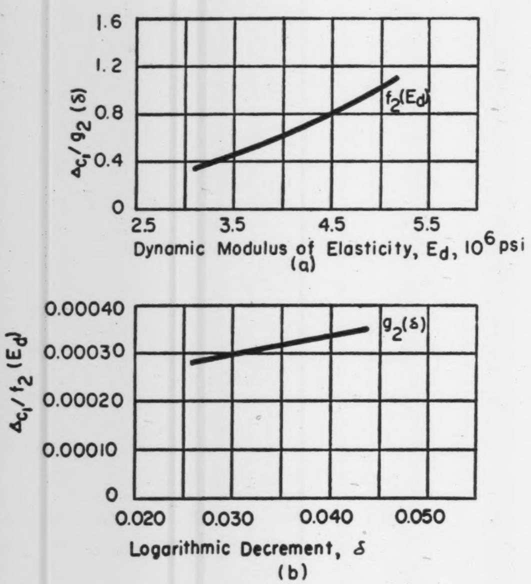

FIG. 12.—Functions of Δ_{c1} for Creep in Flexure.

where:

Δ_c = center creep deflection of the beam,

Δ_{c_1} = center creep deflection at t_c = unity, and

t_c = creep time.

By applying dimensional analysis and taking in account the constant dimensions and loading conditions of the beam the following is obtained.

$$\left.\begin{aligned}\Delta_{c_1} &= f_2(E_d) \times g_2(\delta)\\ n_{\Delta_c} &= f_2'(E_d) \times g_2'(\delta)\end{aligned}\right\} \quad \ldots\ldots\ldots (12)$$

This equation is obtained by assuming that the concrete strength is completely defined by the sonic properties.[4]

Again using the step-by-step approximations and grossing of errors methods, the functions f_2, g_2, f_2' and g_2' are found statistically and are shown in Figs. 12 and 13. The agreement of predicted and experimental results is shown in Fig. 14.

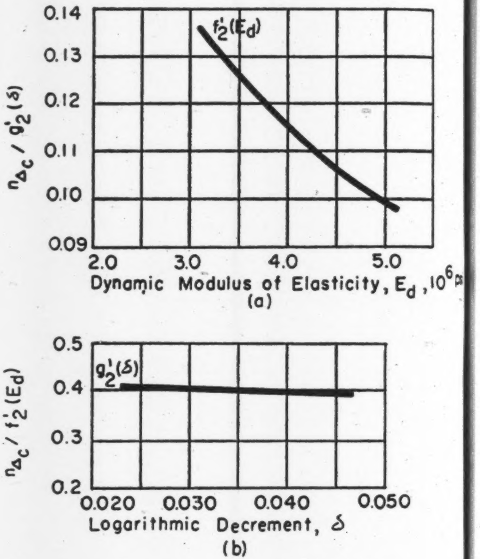

FIG. 13.—Functions of n_{Δ_c} for Creep in Flexure.

Creep of Beams with Concentrated Loads:

Two 6 by 6 by 64-in. beams were loaded at their third points while simply supported. The logarithmic plots of the data obtained again showed that the creep deflections of the beams could be represented by power functions of the creep time ($\Delta_c' = \Delta_{c_1}' t_c^{\,n_{\Delta_c}}$). The creep strains along the depth of the beams again showed a faster creep rate in the tension area. This phenomenon was also indicated by the steeper slopes of the straight lines of the creep-deflection curves as compared to those of their companion cylinders when subjected to axially compressive stresses.

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[4] This is not exactly true and is discussed in reference (5).

It is obvious that with such a small amount of test data, no valid statistical study on the direct relationships between the coefficients Δ'_{c1} and n_{Δ_c} and the sonic properties of their companion cylinders can be made as was done for the uniformly loaded beams. Therefore, these data were instead related to the values of Δ_{c1} and n_{Δ_c} obtained from Eq 12. These values were then corrected to the beams under discussion by a very simple procedure.

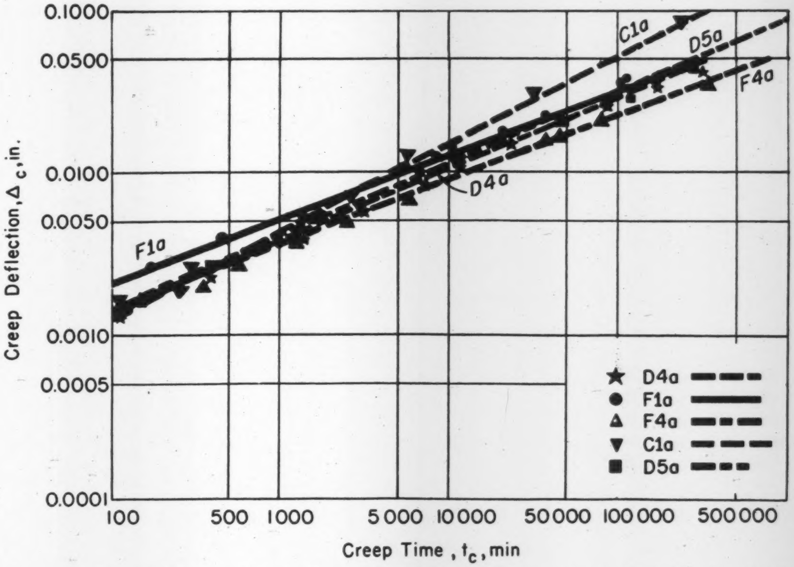

FIG. 14.—Comparison of Predicted Creep Deflection and Actual Creep Deflection for Uniformly Loaded Beams.

For an elastic beam under load, its center deflection will be indicated by an expression of the following nature:

$$\Delta_{c_e} = K/E \qquad (13)$$

where:

Δ_{c_e} = elastic mid-span deflection,

E = modulus of elasticity of the material of the beam, and

K = a constant depending upon the dimensions of the beam and the loading conditions on the beam.

For a viscoelastic beam, it is assumed that the center deflection of the beam can still be represented by Eq 13, only now the term E is not exactly the modulus of elasticity but rather an equivalent modulus which depends on the rheological properties of concrete and the creep time. If the beams under consideration were loaded to less than 50 per cent of their maximum strength, it can be assumed that the rheological properties are independent of the loading conditions (that is, the creep of concrete is proportional to the sustained stress). This, therefore, means that the equivalent moduli of elasticity of two concrete beams are the same if the creep times in consideration are the same for the two beams and if the beams are of the same concrete. The assumption leads to a simple means of relating the creep deflections of a concrete beam to another beam of the same concrete. They are related by the inverse ratio of the constants K and K', which can be determined from elastic analysis:

$$\Delta_c' = \Delta_c \frac{K'}{K} \text{ .... } (14)$$

If Δ_c is the creep deflection predicted for 4 by 6 by 100-in. beams loaded under their own weight while simply supported, and Δ_c' is the creep deflection of 6 by 6 by 64-in. beams under third-point loadings while simply supported, then the ratio of K'/K is found from the ratio of their elastic deflection constants as follows:

$$\frac{K'}{K} = \frac{\dfrac{23}{1296} \times \dfrac{PL'^3}{\dfrac{b'd'^3}{12}}}{\dfrac{5}{384} \times \dfrac{w \times L^4}{\dfrac{bd^3}{12}}} = 0.000514P \text{ .. } (15)$$

where:

P = concentrated load on beam,
L' = span of beam with concentrated loads,
b' = width of beam with concentrated loads,
d' = depth of beam with concentrated loads,
w = uniform load on beam,
L = span of uniformly loaded beam,
b = width of uniformly loaded beam, and
d = depth of uniformly loaded beam

Substituting Eq 15 in Eq 14, the following equation results:

$$\Delta_c' = 0.000514\Delta_c = 0.000514\Delta_{c_1} t_c^{n\Delta_c} \text{ .. } (16)$$

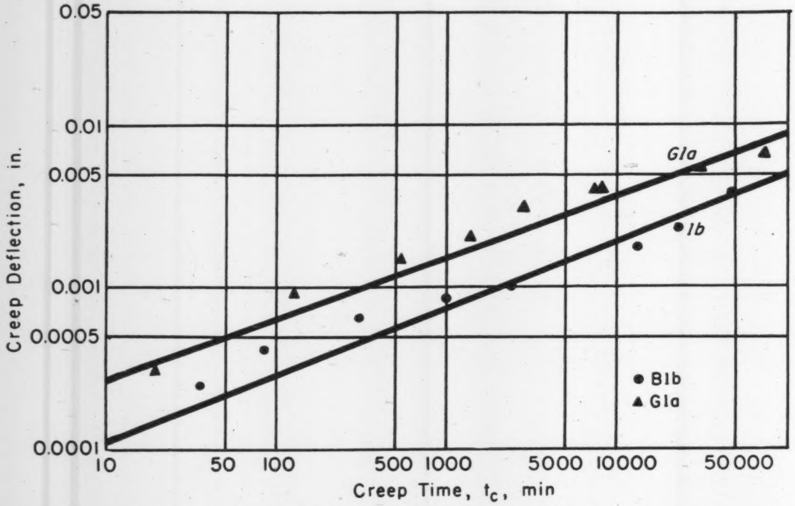

FIG. 15.—Comparison of Predicted Creep Deflection and Actual Creep Deflection for Beams Loaded at the Third-Points.

The creep curves for the two beams tested were then determined from Eq 16, which was found to predict the actual creep data of the beams reasonably well as shown in Fig. 15.

This seems to strengthen the assumptin made in saying that the equivalent moduli of concrete beams are the same at the same creep time, provided they are made of the same concrete and that they are not loaded to more than 50 per cent of their ultimate capacities.

SUMMARY

The results of this study indicate that there is a relationship between the static and dynamic behavior of concrete. They also indicate that this relationship may be represented by a non-linear rheological model, in particular the Maxwell liquid with a stress-softening spring and a time-thickening dashpot. Such a model will adequately describe both creep and relaxation in compression.

In addition to time, the results indicate that magnitude of creep depends on the ratio of sustained stress to ultimate strength, the ratio of dynamic modulus to ultimate strength and the logarithmic decrement. For cylinders loaded to less than 50 per cent of the ultimate strength, the creep is approximately proportional to the percentage of sustained stress (Fig. 4). The relaxation of load in compression of a cylinder held under constant axial strain was found to be a function of the ratio of initial stress to ultimate strength and the logarithmic decrement and is, apparently, independent of the ratio of dynamic modulus to the ultimate strength. The data for relaxation are too few to make definite conclusions.

The behavior of concrete under sustained flexural loading was found to be much more complicated than the behavior under sustained axial compression. This is because of, first the strain gradient and, second, the different magnitudes of creep in tension and in compression. The last item introduces further non-linearity to the creep behavior of concrete and also causes a shifting of the neutral surface which results in an increased creep rate for concrete in flexure. The creep in tension may be as much as three times the creep in compression for specimens stored and tested in a constant temperature and humidity room. All flexure specimens were at least 23 days old before testing.

Although the data available for creep in flexure are meager and are only for two different types of beams there is indication that the creep deflection curve of one type can be predicted from the creep curve of another provided both beams are loaded to less than 50 per cent of their ultimate strength.

The advantages of being able to predict long-time behavior of concrete from tests taking only a few minutes are exciting to consider. The designer might be provided with more complete information on the concrete to be used in such structures as dams and arches. This would result in a better and more economical design.

Further study is needed to determine the feasibility of using the methods described for predicting the behavior of different concretes under different environments and loadings. No doubt the methods suggested can be refined or modified to produce better results.

Acknowledgment:

This investigation was conducted in Talbot Laboratory at the University of Illinois as a project of the Engineering Experiment Station. The authors wish to thank A. M. Freudenthal of the Department of Civil Engineering of Columbia University for his many useful suggestions. This paper is based on the doctoral dissertation of the senior author.

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THE EFFECT OF WEATHERING ON CERTAIN MORTARS EXPOSED IN BRICK MASONRY WITH AND WITHOUT CAPS AND FLASHINGS*

By J. W. McBurney[1]

Synopsis

An exposure test plot was established at the National Bureau of Standards in 1936 for the purpose of observing the effect of weather on various masonry specimens and materials. As a part of this program there was included a set of 16 small walls all built from the same brick but differing in the composition of mortar (four types) and also in the use of caps and flashings to give four types of protection. Photographs were made of these wallettes shortly after completion in 1936 and some of the wallettes were again photographed in 1948 and others in 1952. This paper presents a selection of these photographs and considers the durability of the several mortars as affected by the use of caps and flashings.

Prior to 1936 a considerable number of laboratory freezing-and-thawing tests had been made at the National Bureau of Standards and elsewhere on individual brick units, and a few tests were reported on mortar specimens cast in non-absorbent molds and on small brick-mortar assemblages. Differences of opinion had arisen both with respect to suitable techniques for making laboratory freezing-and-thawing tests and the relation of such tests to the behavior of masonry structures exposed to natural weathering. An exposure test plot was therefore established on the grounds of the National Bureau of Standards. On this plot during the fall of 1936, 125 small piers or wallettes were built for the purpose of comparing their behavior under weathering as masonry with the results of laboratory tests on the constituents of the masonry. As part of this program there was included a set of 16 wallettes designated at that time as series B and all built from the same type of brick but differing in the composition of the mortars (4 types ranging from 1:3 lime putty - sand to $1:\frac{1}{4}:3$ portland cement - lime - putty - sand by volume) and each type of mortar having 4 types of protection ranging from none to a combination of a metal cap on top of the wallettes with a metal flashing through the mortar bed joint just above ground level. Photographs were made of the wallettes shortly after their completion in 1936 and some of the wallettes were again photographed in 1948 and others in 1952. A selection of these photographs is presented and discussed subsequently. This paper discusses the observations made during the outdoor exposure tests.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

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Materials

A side-cut clay brick made in Washington, D. C., was selected for construction of the 16 wallettes. The properties of the brick used were as follows: compressive strength 4120 psi average with maximum and minimum of 7900 and 3630 psi, respectively; water absorptions by 24-hr cold immersion were 8.7 12.4, and 4.2 per cent (average, maximum, and minimum); the coresponding absorptions by 5-hr boiling were 12.3, 16.0, and 7.3 per cent; the derived saturation coefficients averaged 0.76 with a range of 0.66 to 0.80; the initial rates of absorption ranged from 12 g to 35 g averaging 23 g per 30 sq in. These bricks, when received, showed some kiln cracking on their faces. Exposure tests of individual brick, stood on end and half buried in the ground, showed no evidence of disintegration in the period 1936 to 1952. No efflorescence was developed by 7-day wick tests.

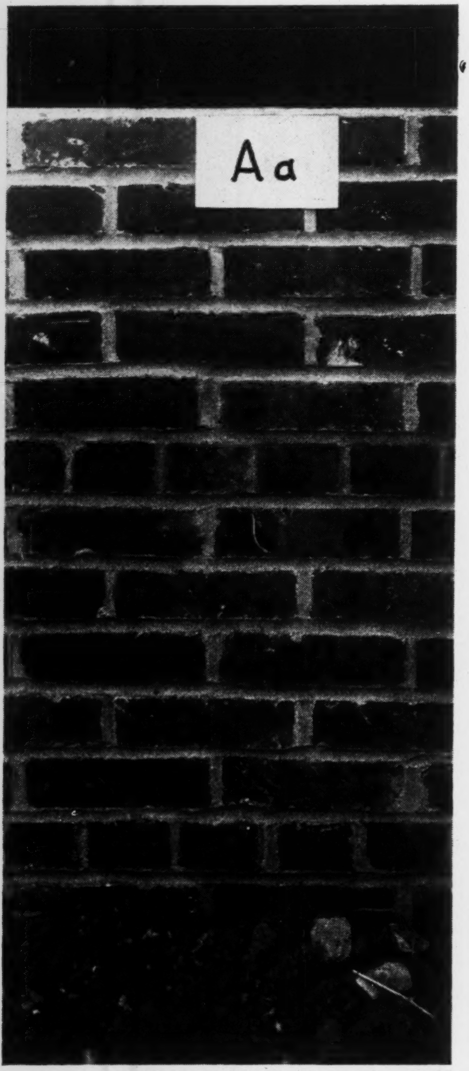

Fig. 1.—Wallette With No Protection Laid With Type A Mortar and Photographed in 1936.

1:1/4:3 cement-lime-sand, by volume.

Since a discussion by Mr. E. C. Shuman[2] compares the limitations on mortar other than strength given in various building codes, the designations A, B, C, and D for these mortar proportions are used since the codes considered by Mr. Shuman use these identifications. These four mortars A, B, C, and D, corresponding to the designations M, N, O, and K, respectively, of ASTM Specification C 270[3] were limited in composition to mixtures of portland cement, lime putty made by slaking pulverized high-calcium quick lime, and Potomac River masons sand. The cement was Government tested material conforming to Federal Specification SS-C-191. The lime was the lime *A* described by Wells, Bishop, and Watstein (**1**)[4] and was used in certain mortars reported by Fishburn, Watstein, and Parsons (**2, 3**) and by McBurney, Copeland, and Brink (**4**). The Potomac River sand did not differ significantly in grading from that reported previously (2, 3, 4) and, since the proportions of cement, lime, and sand were essentially the same, the properties of the mortars used in these wallettes can be assumed to be the same as those reported in the above references. Reference is also made to the paper by McBurney (4) (Fig. 1) for the frequency distributions of test results on mortars A, B, and C. It can be stated that all of the mortars used in the wallette here reported conformed to the proportion specification of American Standard Building Code Requirements for Masonry (A41.1—1944 (5)). The weight proportions were 1:0.10:3, 1:0.43:5, 1:0.85:7.5 cement-lime putty-sand and 1:6 lime-sand by weight calculated on the basis of dry materials (lime solids in the putty and dry sand). The weight proportions were intended to give 1:$\frac{1}{4}$:3, 1:1:6, 1:2:9, and 1:3 proportions, by volume, conforming to the A, B, C, and D mortars as then defined. The consistency of the mortar was adjusted in accordance with the mason's wishes.

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[2] See p. 1283.

[3] Tentative Specification for Mortar for Unit Masonry (C 270 – 54 T), 1955 Book of ASTM Standards, Part 3, p. 427.

[4] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1283.

CONSTRUCTION

The 16 wallettes were built 4 ft high, 3 ft long, and 8 in. in nominal thickness. Trenches 1 ft in depth were dug and the wallettes were built on the compacted soil thus exposed. Four types of protection afforded by caps and flashings were involved as follows: *a* no protection; *b* the top covered with a copper cap; *c* a through copper flashing inserted in the bed joint one brick course above ground level; and *d* both a copper cap and flashing. Photographs of *a* and *d* types are given in the next section. After completion, the trench was back filled.

These wallettes were built by the same mason who had been employed to construct some of the large walls reported in reference (6) and the same workmanship was used. Quoting from that report "characteristics of the work were that there was practically no mortar in the longitudinal vertical joints, and horizontal mortar beds were deeply furrowed, and the brick were laid at a high rate." Also characteristic of this mason's work

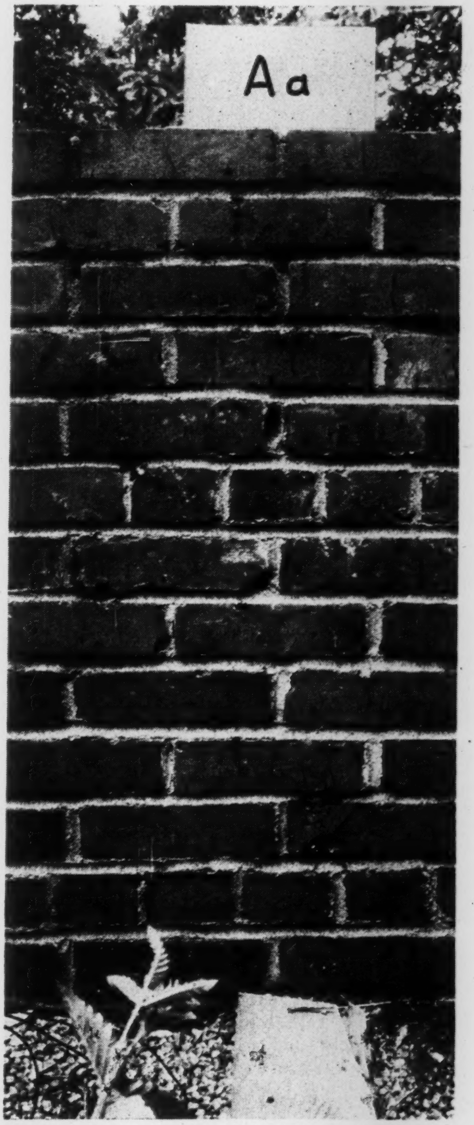

Fig. 2.—Same Wallette Shown in Fig. 1, Photographed in 1948.

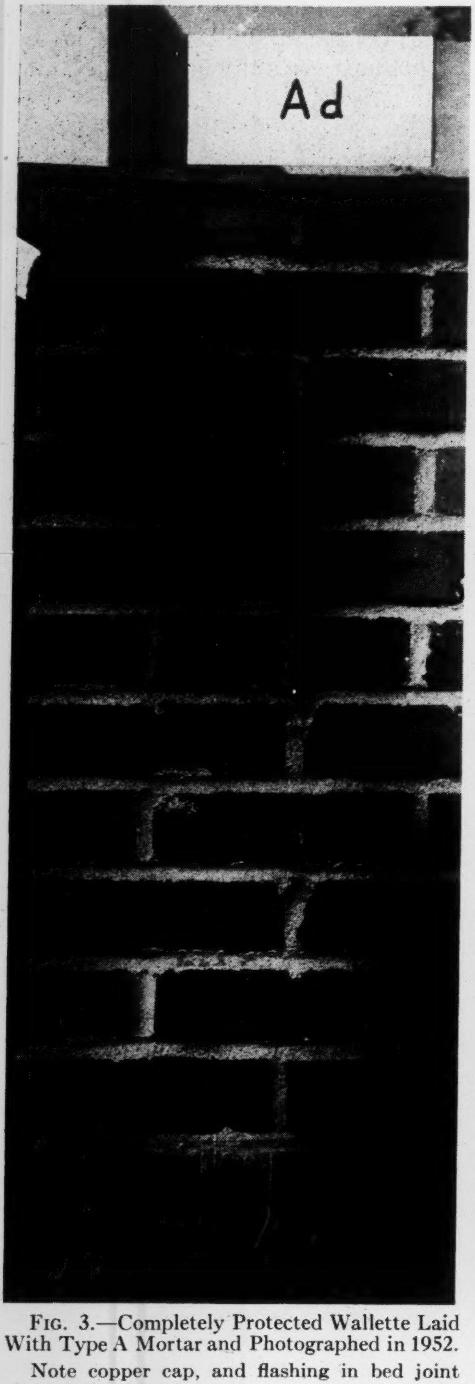

FIG. 3.—Completely Protected Wallette Laid With Type A Mortar and Photographed in 1952.

Note copper cap, and flashing in bed joint above ground level.

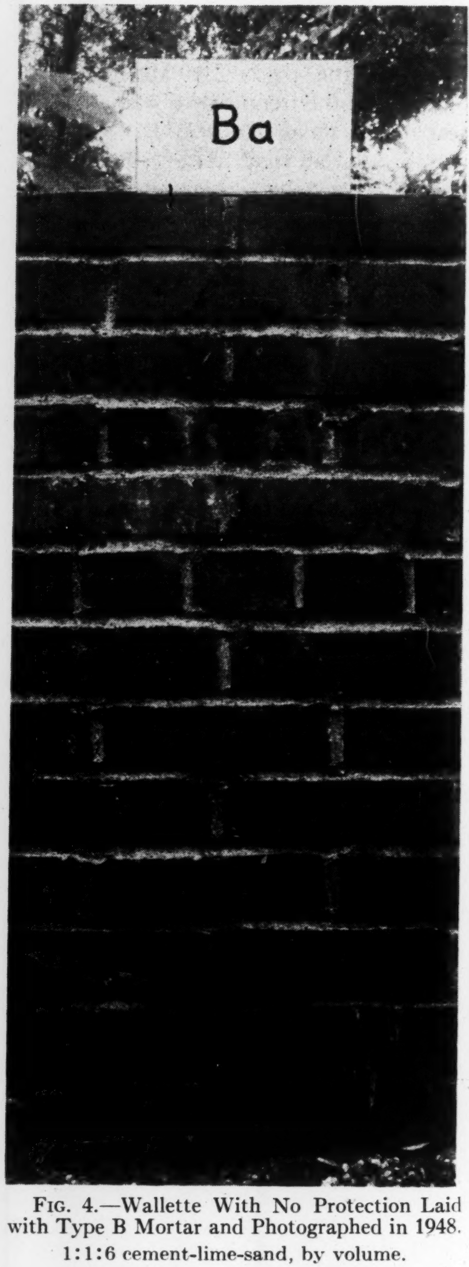

FIG. 4.—Wallette With No Protection Laid with Type B Mortar and Photographed in 1948.

1:1:6 cement-lime-sand, by volume.

was that the only mortar in the vertical joint between the ends of the bricks was that cut from the bed joints. Tooling of the mortar joints was done on the east face of the wallette. The construction was completed in the period September 17 to October 16, 1936. In 1939 a topping of gravel was added to the test plot which in some instances brought the ground level up to or slightly above the flashings in the *c* and *d* wallettes.

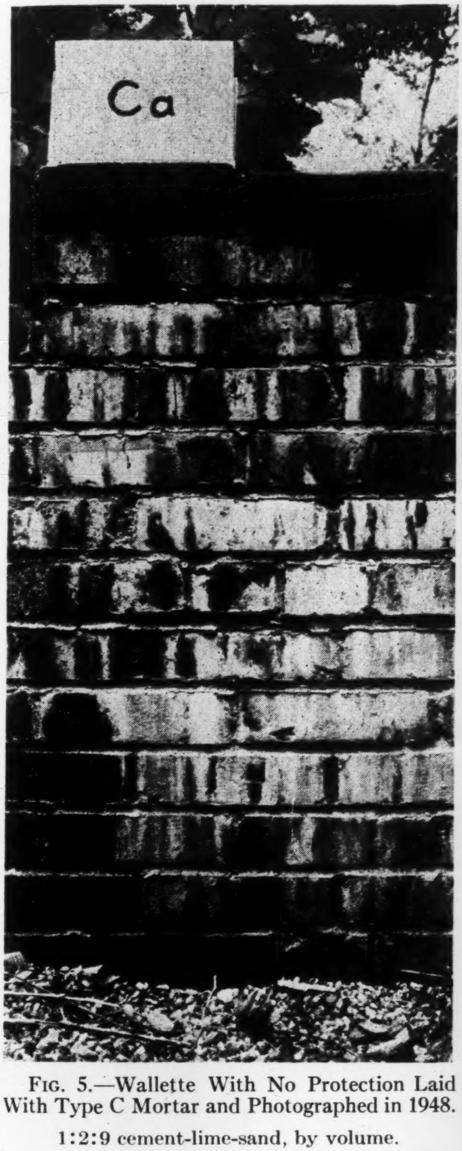

FIG. 5.—Wallette With No Protection Laid With Type C Mortar and Photographed in 1948.

1:2:9 cement-lime-sand, by volume.

EXPLANATION AND DISCUSSION OF PHOTOGRAPHS

The cards originally photographed had identified the series B and the mortars as 4, 3, 2, and 1 corresponding to the A, B, C, and D described below. The symbols for type of protection, *a*, *b*, *c*, and *d* were unchanged. The cards shown in the current photographs identify the mortar compositions as A ($1:\frac{1}{4}:3$ cement - lime - sand, by volume), B (1:1:6) C (1:2:9) and D (1:3 lime - sand). The letters, *a*, *b*, *c*, and *d* refer to the type of protection described in the preceding section. The *b* and *c* protections are, however, not shown in the photographs reproduced in this paper. The *a* and *d* types of protection are referred to in the captions as: "no protection" and "completely protected." The dates in the captions, 1936, 1948, or 1952 refer to the dates at which the photographs were taken. More exactly these dates were November 26, 1936, August 23, 1948, and July 23, 1952. The order of presentation of these illustrations starts with mortar A through to mortar D.

Figures 1 and 2 illustrate the effect of a 12-yr exposure on an unprotected wallette laid with Mortar A ($1:\frac{1}{4}:3$). Except for enlargement of the surface cracking on the bricks, no effect is visible. The whitening of the mortar joints varied with changes in weather. Figure 3 illustrates the effect of a 16-yr exposure on a completely protected wallette also laid with type A mortar. Note the flashing in the lowest visible bed joint. As with Fig. 2, no effect from exposure is noted. In general, the mortar joints in capped wallettes (*b* and *d*) were whiter after exposure than those on uncapped

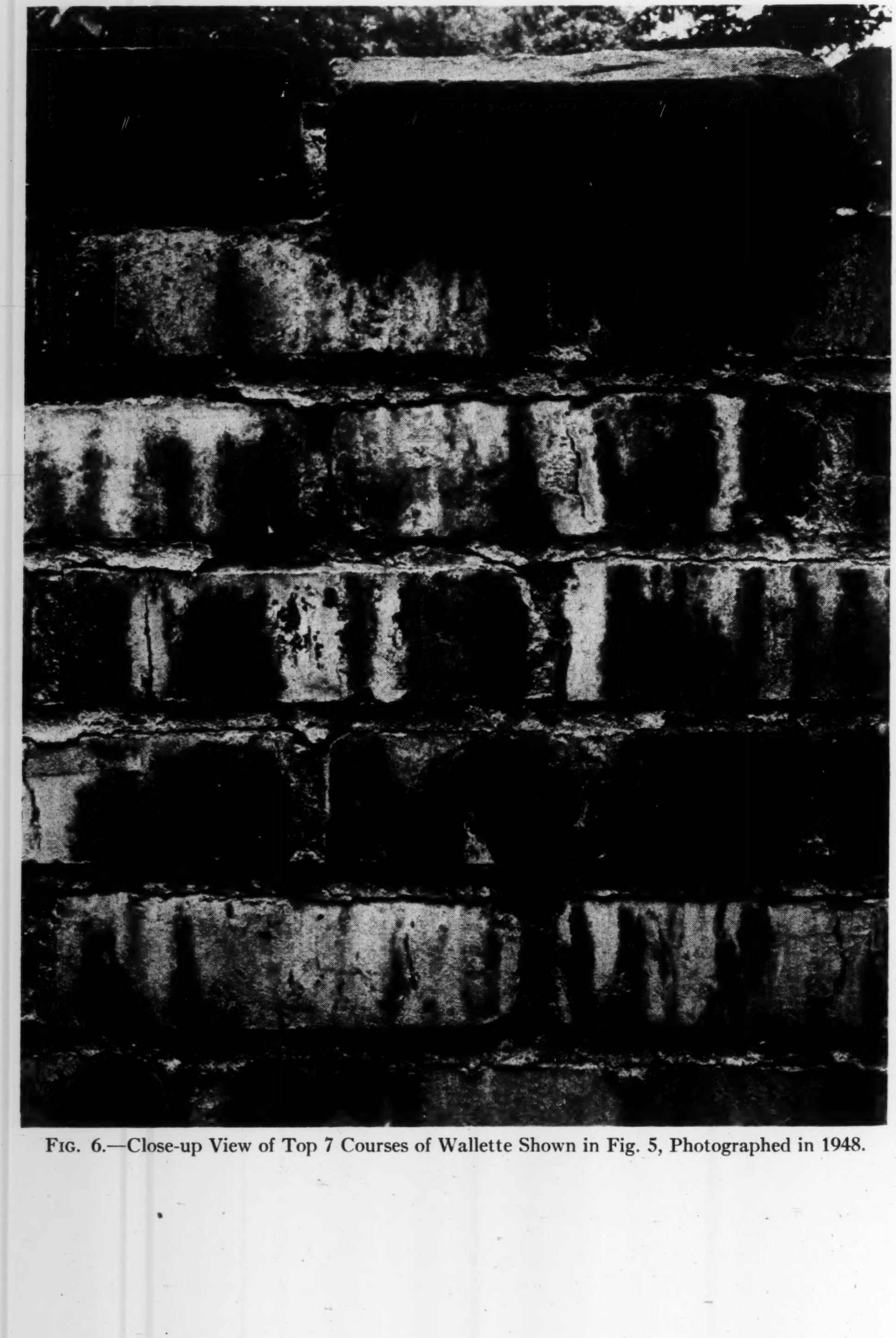

Fig. 6.—Close-up View of Top 7 Courses of Wallette Shown in Fig. 5, Photographed in 1948.

wallettes (*a* and *c*). Since all of the 1936 photographs were indistinguishable from Fig. 1 except for their labels and differences in protection, these pictures, with two exceptions, are omitted. Figure 4 shows the 12-yr exposure of an unprotected wallette laid with type B (1:1:6) mortar. Except for traces of smeared mortar on the faces of certain bricks, Fig. 4 is indistinguishable from Fig. 2.

Figure 5 shows the results of 12-yr exposure on an unprotected wallette laid

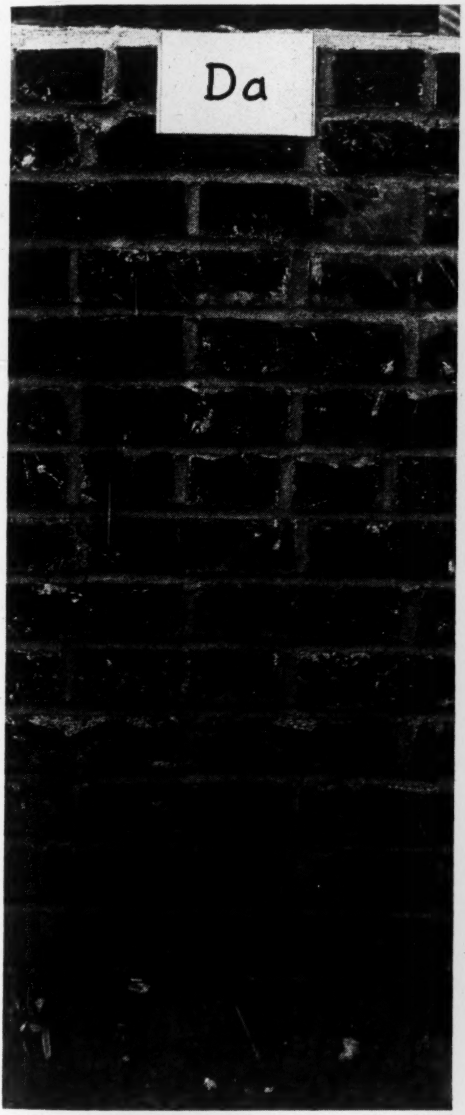

Fig. 7.—Wallette With No Protection Laid With Type D Mortar, Photographed in 1936. 1:3 lime-sand, by volume.

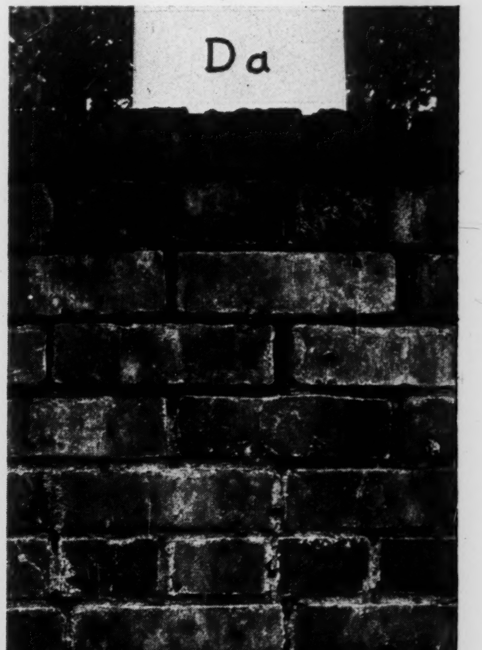

Fig. 8.—Same Wallette Shown in Fig. 7, Photographed in 1948.

with type C mortar (1:2:9). Figure 6 is a close-up of the upper part of the same wallette. Note the loosening of the mortar surfaces on the top three bed joints, the detachment of the top course of brick, and the coating of calcium carbonate film over brick surfaces. The first two effects were seasonal. In the warm months the mortar would form a thin brittle film on its exposed surface but

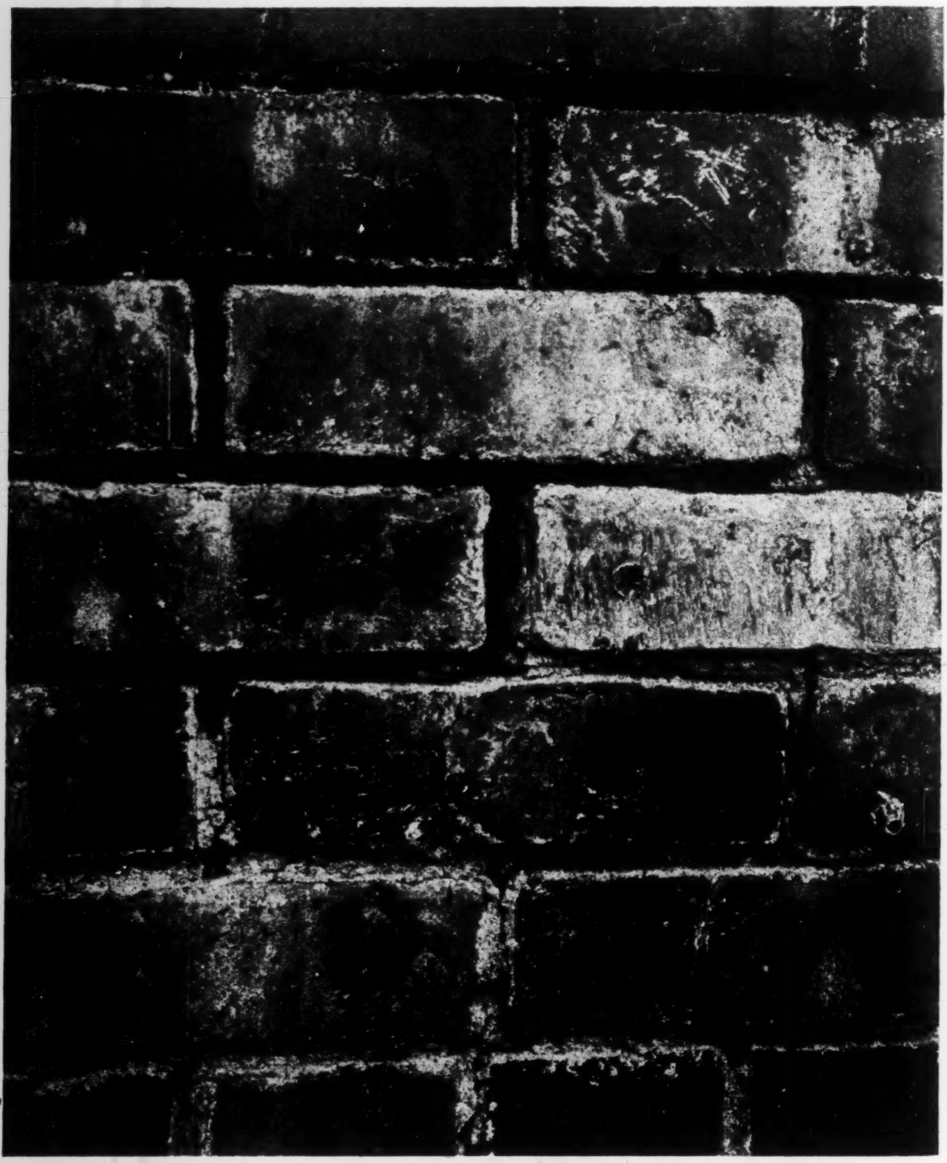

Fig. 9.—Close-up View of Upper Portion of Wallette Shown in Fig. 8, Photographed in 1948.

subsequent freezings and thawings would loosen this surface from the unhardened mortar base. At the end of the first winter, the bond of the top brick course to the mortar bed had failed as evidenced by lack of resistance to slight manual lifting, but during the following summer most of the bricks had recemented (autogenous healing). The bricks again became loose during the second winter but recementing ceased after the third winter. All wallettes laid with type A or B mortars showed no bond failures through the entire time of exposure. The coating shown in Fig. 5 was identified as calcium carbonate by its active effervescence with hydrochloric acid and its insolubility in water. This wall was built on September 17, 1936. On September 24, a total precipitation of 0.07 in. was recorded by the Weather Bureau and on September 30, midnight to midnight, it rained 1.45 in. The deposit on the surface shown in Fig. 5 developed shortly thereafter.

Figure 7 shows an unprotected wallette laid with type D mortar (1:3 lime - sand) as it appeared in 1936. Figure 8 is the same specimen as it appeared in 1948. A close-up of the upper six courses is shown in Fig. 9. Figures 8 and 9 show extreme disintegration of mortar compared with the relatively moderate attack by weathering illustrated in Figs. 5 and 6. Note the almost complete disappearance of mortar in the vertical joints in the top courses and also below, close to ground level. This is explained by the mason's practice of buttering the outside vertical edges of his stretchers using only mortar cut from the bed joint. Filling of vertical joints to less than $\frac{1}{2}$ in. back from the face can result from this technique. The locations at the top and bottom of the exposure obviously are most likely to be saturated with water. The portion of the wall between these top and bottom sections was relatively unaffected. The beneficial effect on durability of keeping water out of the masonry is most strikingly shown by Fig. 10. This wallette, also laid with type D mortar but completely protected (*d*),

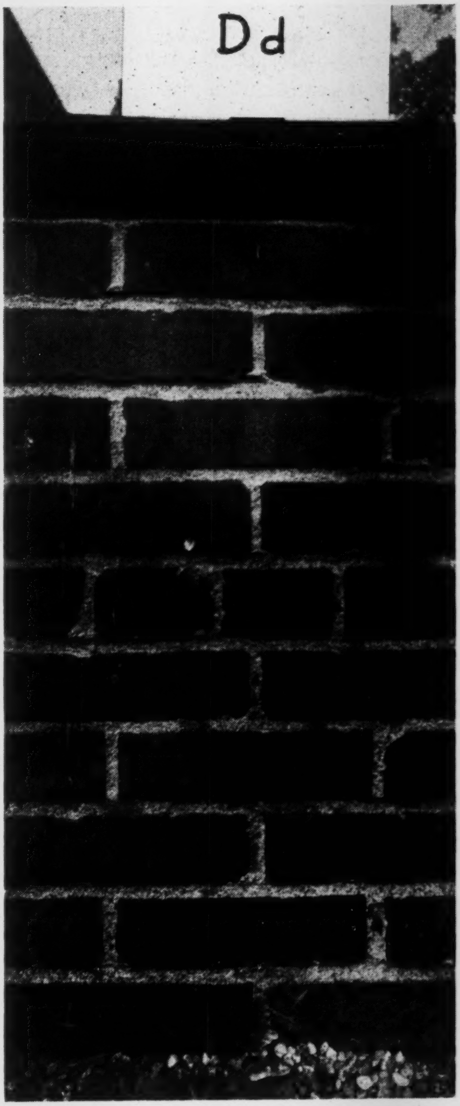

FIG. 10.—Completely Protected Wallette Laid With Type D Mortar, Photographed in 1952.

1:3 lime-sand, by volume. The flashing is concealed by the gravel topping. Compare with Fig. 3.

was indistinguishable from three adjacent wallettes laid at the same time (October 16, 1936) with A, B, and C mortar and recorded in 1952. One of these wallettes (A mortar) was previously shown in Fig. 3. Although not visible in Fig. 10 the wallette showed considerable disintegration of its mortar joints below its flashing and immediately above the original ground level. The four completely protected wallettes laid with mortars A (Fig. 3), B, C, and D (Fig. 10) were inspected in January of 1956 (19 yr and 4 months of exposure) and were indistinguishable one from another in appearance of the faces between cappings and flashings.

During the first three years, weathering effects on C and D mortars had developed very nearly to the degree shown in Figs. 5 and 6 and 8 and 9, which represented 12-years of exposure. Temperature records provided no evidence for assuming that weather conditions for the first three years (1936 through 1938) differed significantly from that prevailing during subsequent exposure.[5]

The weathering of wallettes with protections *b* (cap only) and *c* (flashing only) can be summarized as follows: mortars A and B were indistinguishable in appearance from those illustrated in Figs. 2 and 4.

When wallettes laid with C and D mortars were protected by caps only, evidence of weathering was seen only near their bases. These same mortars protected by flashing only (*c*) were attacked much as is shown in Figs. 5 and 8.

Summary and Conclusions

Sixteen wallettes 4 ft high, 3 ft long and 8 in. in nominal thickness were built of one type of brick and three types of portland cement - lime putty mortars in proportions of 1:$\frac{1}{4}$:3, 1:1:6, and 1:2:9 cement - lime - sand and one 1:3 lime - sand all by volume. Protective measures were of four types, no protection, a metal cap, a metal through flashing and both cap and flashing. All wallettes were embedded 1 ft in the ground.

Among the principal observations and conclusions are the following:

1. Where both protective caps and flashings were used, no significant differences were observed in the weathering as rated by visual inspection of the above-ground portions of wallettes, regardless of the mortar used (1:$\frac{1}{4}$:3 cement - lime - sand to 1:3 lime - sand).

2. Where 1:$\frac{1}{4}$:3 and 1:1:6 cement - lime - sand mortars were used, wallettes without caps or flashings showed no effect from weathering exposures for the duration of the exposure (12 to 16 yr).

3. Where 1:2:9 cement - lime - sand or 1:3 lime - sand mortar were used, wallettes without protection showed definite evidence of weathering during the first three years of exposure. Little change was observed during the subsequent 9 to 13 yr.

4. Protection by means of cap only or flashing only were effective in localizing the appearance of weathering damage for 1:2:9 cement - lime - sand and 1:3 lime - sand mortars. On capped wallettes the damage appeared near the base of the wallettes whereas flashed wallettes showed weathering effects little different from those observed on unprotected wallettes.

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[5] The effective weathering exposure conditions in Washington are considered severe for the country at large, excepting those in a few mountainous regions.

REFERENCES

(1) L. S. Wells, D. L. Bishop and D. Watstein, "Differences in Lime as Reflected in Certain Properties of Masonry Mortars," *Journal of Research*, Nat. Bureau of Standards, Vol. 17, Dec., 1936, p. 895–907. (RP 952).

(2) C. C. Fishburn, D. Watstein and D. E. Parsons, "Water Permeability of Masonry Walls," *Building Mats., and Structures Reports, No. 7*, U. S. Govt. Printing Office, Oct. 8, 1938.

(3) C. C. Fishburn, "Water Permeability of Walls Built of Masonry Units," *Building Mats., and Structures Reports, No. 82*, U. S. Govt. Printing Office, April 15, 1942.

(4) J. W. McBurney, M. A. Copeland and R. C. Brink, "Permeability of Brick-Mortar Assemblages," *Proceedings*, Am. Soc. Testing Mats., Vol. 49, p. 1333 (1949).

(5) "American Standard Building Code Requirements for Masonry" Approved Jan. 22, 1944 by the Am. Standards Assn. as American Standard A41.1—1944. (Issued as *Miscellaneous Publication M174* by the Nat. Bureau of Standards.)

(6) A. H. Stang, D. E. Parsons and J. W. McBurney, "Compressive Strength of Clay Brick Walls," *Journal of Research*, Nat. Bureau of Standards, Vol. 3, Oct. 1929, pp. 507–571. (RP 108.)

DISCUSSION

MR. E. C. SHUMAN[1] (*presented in written form*).—The interesting paper by Mr. McBurney, with a little appreciated but simple lesson, recalled the introduction of the writer to such constructional problems in 1923, when tests conducted by the writer over a 2-yr period were reported before the Engineering Society of Wisconsin by Mr. M. O. Withey as "Results of Strength Tests on Mortars for Masonry Construction Cured in Warm and Cold Temperatures."

These results indicated a not unexpected lowering of the strength of mortars when frozen shortly after fabrication. In the subject paper it is shown that strength adequate for structural needs is generally all that is needed for durability in weathering if moisture is kept out of the interior of the wall. This is also illustrated in the accompanying Fig. 11 taken recently of a parapet wall in a residence where mortar failure immediately below the leakable coping is evident. No such distress was noted under the eaves of the adjacent wall of the house which was laid in the same kind of mortar.

Since quality of mortar above some point not defined here is not necessary, it is reasonable to ask whether building codes contain limitations on mortars with respect to durability which are restrictive if adequate protection against the occurrence of moisture within the wall is provided.

A review of certain of the building code requirements discussed at length with Mr. McBurney are of interest.

The first model code known to the author or the writer was the National Board of Fire Underwriters' code of 1905[2] which listed three mortars: (1) a 1:3 portland cement - sand, by volume, with the provision that "this shall not prohibit the use in cold weather of a small proportion of lime to prevent the mortar from freezing"; (2) a 1:1:6 cement-lime putty - sand mortar, by volume; and (3) a 1:4 lime putty - sand mortar by volume. The principal limitations on use were "All foundation walls, isolated piers, parapet walls and chimneys above roofs shall be laid in cement mortar and all other walls built of brick or stone shall be laid in lime and cement mortar mixed."

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[1] Director, Research and Engineering, Kaylo Division, Owens-Illinois, Toledo, Ohio.

[2] "Building Code Recommended by the National Board of Fire Underwriters," 1905 Edition, 85 John St., New York 36, N. Y.

The fourth edition (1920)[3] of this code was unchanged from the first edition except that a 15 per cent replacement of the portland cement by hydrated lime was permitted. Additional limitations included the requirement of cement masonry for arches over doorways and windows and also "hollow block walls shall be laid in portland-cement mortar." The definition of hollow brick included both concrete block and structural clay tile which are referred to as "terra cotta block."

The "Recommended Minimum Requirements for Masonry Wall Construction"[4] of the Department of Commerce, Building Code Committee (1924) provided the following new features: (1) the recognition of 1:3 natural cement-sand mortar as the equivalent of 1:1:6 portland cement-lime-sand mixes; (2) a special 1:1:4 portland cement-lime-sand mortar accepted for masonry built of hollow units; (3) the permissible use of 1:1:6 mortars for foundation walls not less than 12 in. in thickness with the added provision (in appendix) that foundations "should be waterproofed with cement plaster or other effective means"; and, (4) the relations of allowable working stresses to gradings of strengths of units laid with the various types of mortar. The 1931 modifications of these requirements revised upward the allowable working stresses for brick masonry. These values have been incorporated, with a few minor changes, into nearly all subsequent masonry standards. An outstanding feature of the Depart-

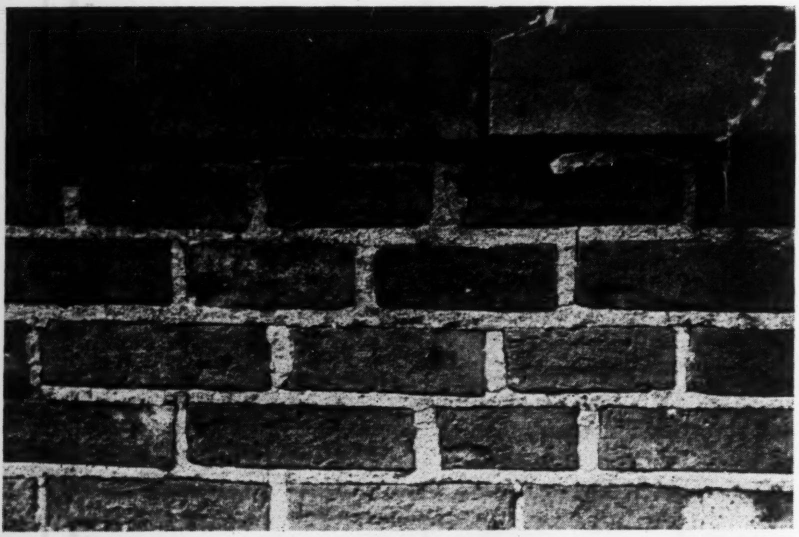

FIG. 11.—Mortar Failure Beneath Leakable Coping of Parapet Wall.

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[3] "Building Code Recommended by the National Board of Fire Underwriters," 4th Edit., Revised Reprint (1920).

[4] "Recommended Minimum Requirements for Masonry Wall Construction," *Report of Building Code Committee*, June 26, 1924. (See also "Modifications" issued March 16, 1931.)

ment of Commerce Masonry Code was an appendix which summarized much of the then available data on masonry and masonry materials and gave reasons for various provisions and requirements.

The first edition (1944) of the American Standard Building Code Requirements for Masonry (A 41.1—1944)[6] may be regarded as an elaboration of the 1924 Department of Commerce code including its 1931 modifications. The 1944 code was the first to include specifications for the several types of mortar which went beyond their definitions in terms of proportions of cement, lime and sand. This specification for mortar was in essence ASTM Tentative Specification C270 - 51 T[5] and included the choice of either property or proportion requirements. The designations (A, B, C, and D) of the mortar types recognized in the 1944 code have been universally followed in all subsequent model codes insofar as designations other than descriptions are used. For this reason A (now A-1 and A-2), B, C, and D are used in the subject paper instead of the corresponding M, S, N, O, and K designation presented in Specification C 270 - 54 T.[7] The 1944 code was the first to recognize type II and type I masonry cement in 1:3 cement - sand proportions as the equivalents of 1:1:6 and 1:2:9 portland cement - lime - sand mortars. The mortars designated as A, B, C, and D in the subject paper and in the 1944 code are defined as 1:$\frac{1}{4}$:3, 1:1:6, 1:2:9 portland cement - lime - sand and 1:3 lime - sand proportions, by volume, and are not referred to as masonry cement mortars. A, B, and D will be recognized as the equivalents of the three mortars discussed in the preceding references up to 1944.

The restrictions on the use of mortar in this code are quoted below:

> "Types of Mortars Required—Masonry shall be laid in type A, type B or type C mortar, except as follows:
>
> "(*a*) Type A mortar shall be used in nominal 10-in. cavity walls, foundation walls of hollow masonry units, and masonry linings of existing masonry walls.
>
> "(*b*) Type A or type B mortar shall be used in footings, foundation walls of solid masonry units, isolated piers, load-bearing or exterior walls of hollow masonry units, hollow walls of masonry, and cavity walls exceeding nominal 10-in. thickness.
>
> "(*c*) Type D mortar may be used in solid masonry walls, other than parapet walls or rubble stone walls, not in contact with the soil and not less than 12 in. thick nor more than 35 ft in height, provided the walls are laterally supported at intervals not exceeding 12 times the wall thickness."

The 1953 revision of A 41.1[8] is very little changed from the 1944 code. The specification for mortar in the original edition is now replaced by reference to ASTM Designation C 270 - 52 T.[9] The 1944 and 1953 standards are identical with respect to types of mortar permitted with the exception that the A mortar of A 41.1—1944 is now replaced by A-1 (the original A) and A-2 (1:$\frac{1}{2}$:4$\frac{1}{2}$) and the requirements for types of mortar permitted (previously quoted) are replaced by a table.

This table is incorporated in the 1955 edition of the National Board of Fire

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[5] Tentative Specification for Mortar for Unit Masonry (C 270 - 51 T), 1951 Supplement to Book of ASTM Standards, Part 3, p. 153.

[6] "American Standard Building Code Requirements for Masonry" approved Jan. 22, 1944 by the American Standards Association as *American Standard A 41.1—1944.* (Issued as Miscellaneous Publication M 174 by the National Bureau of Standards.)

[7] Tentative Specifications for Mortar for Unit Masonry (C 270 - 54 T), 1955 Book of ASTM Standards, Part 3, p. 427.

[8] "American Standard Building Code Requirements for Masonry" approved Sept. 28, 1953 by the American Standards Association as *American Standard A 41.1—1953.* (Issued as Miscellaneous Publication M 211 which superseded M 174 above.)

[9] Tentative Specifications for Mortar for Unit Masonry (C 270 - 52 T), 1952 Book of ASTM Standards, Part 3, p. 383.

Underwriters National Building Code[10] and is the golden anniversary of their 1905 edition.[2]

The 1953–54 Revision of the Southern Standard Building Code[11] in Section 1402.17 presents without change the material on "types of mortar required" given in the 1944 edition of A 41.1.[5]

The Basic Building Code (1955)[12] of the Building Officials Conference of America is unusual in that there are few limitations on the use of the three types of mortars mentioned in this standard. These three types of mortars are "cement" (1:$\frac{1}{4}$:3), cement lime (1:1:6) and lime. The lime mortar includes proportions from 1:3 lime-sand to 1:2:9 cement-lime-sand. Cement or cement-lime mortars are required for masonry unit footings. Appendix K-1 of this code follows in part material in A 41—1953 and like that standard, omits values for allowable compressive strength for masonry built of hollow units with lime mortar.

The first edition of the Uniform Building Code of the Pacific Coast Building Officials Conference was issued in 1927. The 1943 edition recognized only two cement-lime mortars which were 1:$\frac{1}{4}$:3 and 1:$\frac{1}{2}$:4$\frac{1}{2}$ cement-lime-sand, by volume, respectively. The next edition (1946) lists these two mortars now called A and B, and a third mortar which could be either 1:1:6 or one part type II masonry cement to 3 parts sand, by volume. From the table of working stresses in unreinforced masonry it may be concluded that with the exception of grouted masonry the code had no limitations other than strength with reference to choice of mortars. The 1955 edition[13] combines A and B of the 1946 code into one mortar (A) and defines B as 1 part portland cement to $\frac{1}{2}$ to 1 parts lime and the usual 3 parts of sand to the sum of the cement and lime. The 1:3 type II masonry cement-sand mortar is now called C and has the same minimum compressive strength at 28 days as the B mortar. A new mortar D is defined as 1 cement, 1$\frac{1}{4}$ to 2 lime and the usual proportion of sand. The limitations are "masonry used in foundation walls and footings shall be laid up in type A mortar. Type D mortar shall be used only in interior non-structural walls."

In terms of the A-1, A-2, B, C, and D mortars of A 41.1—1953[8] the requirements of the five current "model codes" here considered can be summarized with respect to durability requirements of mortars as follows: A-1 and A-2 have no limitations, B mortar is permitted for foundation walls of solid units by A 41.1—1953, the National Code and the Southern Code. The Uniform Code does not permit B mortar for foundation walls and footings and requires A-1 and A-2 mortars for such locations. The same code restricts C mortar to interior use and does not recognize D mortar for any purpose. The Basic Code recognizes A, B, and D mortars and apparently has no restrictions based upon durability.

From the preceding summary and omitting consideration of restrictions on use of types of mortar given in current building codes which are obviously based upon strength or structural requirements, the results of the exposure tests reported by the author are believed to warrant the following comments on the building code requirements. Since the current National,[10] Southern,[11] and American Standard Building Code Requirements for Masonry[8] are alike in their restrictions on mortar, these three standards will not be discussed individually but will be referred to as ASA requirements. Types A-1 and A-2 mortars are combined as A for the purposes of these comments.

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[10] "National Building Code 1955, Golden Anniversary Edition Recommended by the National Board of Fire Underwriters," 85 John St., New York, N. Y.

[11] "Southern Standard Building Code," 1953–1954 Revision, Southern Building Code Congress, Brown-Marx Building, Birmingham, Ala.

[12] "Basic Building Code," (1955) Building Officials Conference of America, Inc., New York, N. Y.

[13] "Uniform Building Code," 1955 Edit., Vol. 1, Pacific Coast Building Officials Conference, 610 South Broadway, Los Angeles 14, Calif.

1. The behavior of A and B mortars (American Standard A 41.1) exposed in brick masonry without protection warrant their use in foundation walls of solid units. The prohibition of B mortar in footings and in foundations, piers, and walls subject to high wind pressure when built of hollow units is based upon strength requirements.

2. The permissible use of C mortar in exterior walls of solid units and D mortar where there are limitations on minimum thickness and where there are more rigid requirements for lateral support appear justified from the behavior of these two mortars in wallettes completely protected *d*. A properly designed exterior wall would be considered to include protection at the tops either by a flashing or by overhanging eaves, and does not come in contact with soil at its base.

3. The restrictions on mortar for parapets other than the prohibition on the use of D mortars for this purpose in the ASA requirements were found in the 1905[2] code. Unless a highly impermeable coping protects the parapet, failure of bond between mortar and units may take place if C mortar is used, and may occur in others as Fig. 11 illustrates.

4. The advice in the Department of Commerce Recommendations[4] that B mortar in foundations be waterproofed by cement plaster is excellent from the standpoint of preventing permeation of water but seems unnecessary with respect to durability.

5. The Basic Code[12] is standard insofar as it provides limitations on the use of mortar. It could be interpreted as permitting D mortar for all uses except masonry unit footings and foundations.

6. The Uniform Code[13] prohibits mortars weaker than B for either structural use or exterior exposure. From the standpoint of durability, this might be unduly restrictive in view of the results on completely protected masonry reported in Mr. McBurney's paper.

Since the subject paper and other inspections indicate the highly beneficial effect of a continuous impervious layer or sheet at the top of masonry walls, particularly parapet walls, it is surprising to the writer that this layer or sheet as a requirement has been overlooked both in codes and in the education of specification writers.

FURTHER DATA ON GAMMA RAY TRANSMISSION THROUGH SOILS*

By R. K. Bernhard,[1] M. Chasek[1] and P. Griggs[1]

Synopsis

Field experiments to determine soil densities by means of radioactive isotopes yielded numerical values with inherent random errors. Statistical analyses are described with the main purpose to determine parameters of the equations which govern the "best fit" lines, that is, correlate soil density, transmitted radiation intensity, and distance between radiation source and detector. Effect of the primary beam is discussed and further studies which seem advisable have been outlined.

Methods of statistical analysis (1)[2] as an engineering research tool are gaining steadily in importance.

Studies with gamma ray transmission to determine soil densities (2,3,4,5) yielded experimental values which, due to the type of measurements involved, contain inherent random errors.

In order to develop so called "best fit" equations, a statistical analysis was made, based on extensive laboratory and field tests.

The paper represents a continuation of experiments described previously (3,4). All field tests are made with improved instruments based on experiences gained from these earlier investigations.

The studies are restricted to 13 different inorganic soils (6) available in the state of New Jersey and usable for various structural purposes.

The main objective of this statistical analysis was to derive the parameters of general equations comprising three significant variables:

1. Soil density, ρ, (wet soil density in lb per cu ft),
2. Transmitted radiation intensity I, in cpm as random variable, and
3. Distance x, in ft between radiation source and radiation detector taken at arbitrarily selected values.

Equations used in the statistical analyses are summarized.

As radiation, source radioactive cobalt (Co^{60}) of approximately 50 millicurie and as a detector, a scintillation crystal (sodium iodide, thallium activated) in combination with a scaler unit were available.

Technique:

Instrumentation.—In the previously described laboratory experiments (3, 4) it was possible to measure the distance x between radiation source and detector

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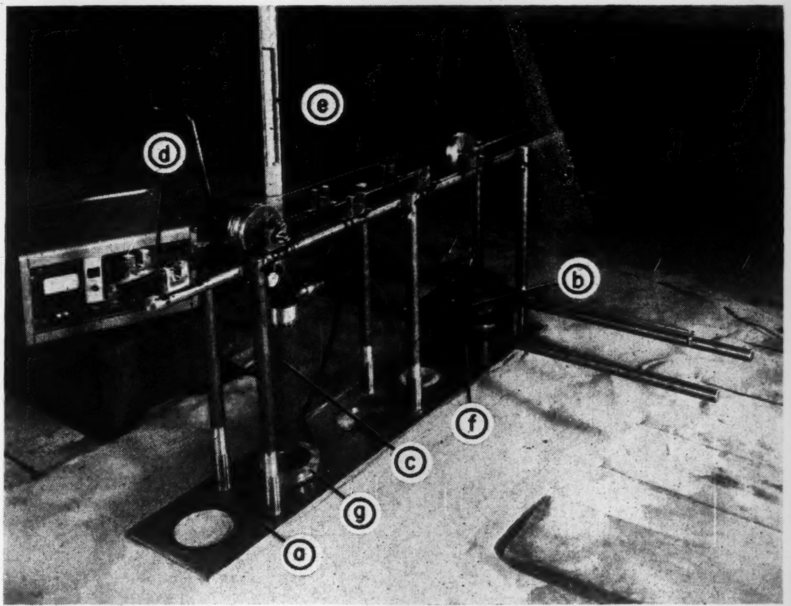
* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] Professor of Engineering Mechanics, Former Research Assistant in Instrumentation, and Former Research Assistant in Civil Engineering, respectively. Rutgers University, The State University of New Jersey, College of Engineering, Bureau of Engineering Research.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1299.



a Bottom plate with 5 holes for 1- 2- 3- and 4-ft source-detector distances.

b Lead container for radiation source.

c Plastic container for detector.

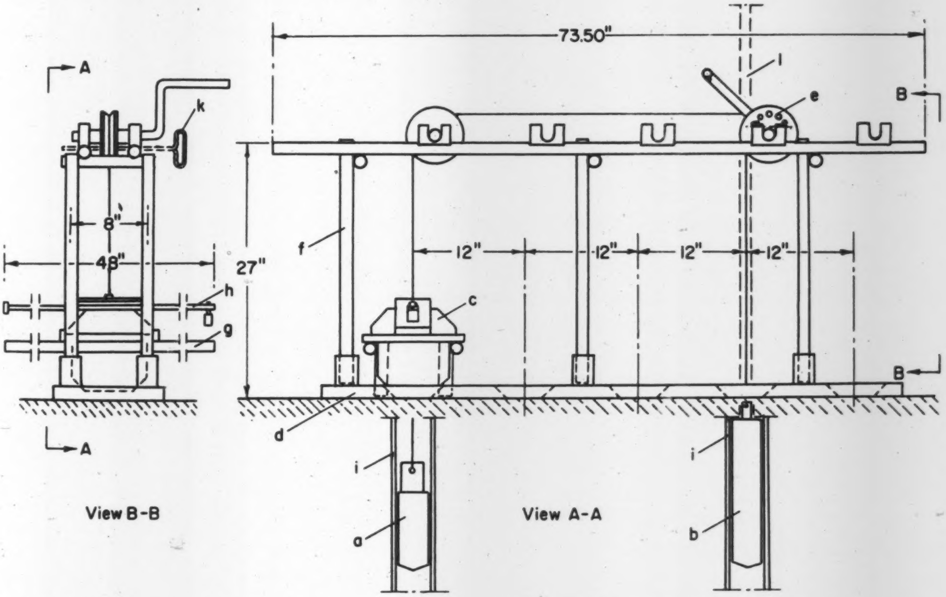
d Counter to register output of detector.

e Scale indicating depth of source and detector below soil surface.

f Access tube inserted to lower radiation source.

g Access tube inserted to lower *c*.

FIG. 1.—Set-up for 3-ft Source-Detector Distance Ready for Lowering Source and Detector to Required Depth below Soil Surface.



a Radiation source container.

b Radiation detector.

c Lead container for *a*.

d Bottom plate.

e Winding mechanism.

f Frame to support *e*.

g Handle bars to move *c* and *a*.

h Bar with padlock to secure *a* in *c*.

i Access tubes for *a* and *b*.

k Bar to hold *a* and *b* at required depth.

l Scale to indicate depth of *a* and *b* below soil surface.

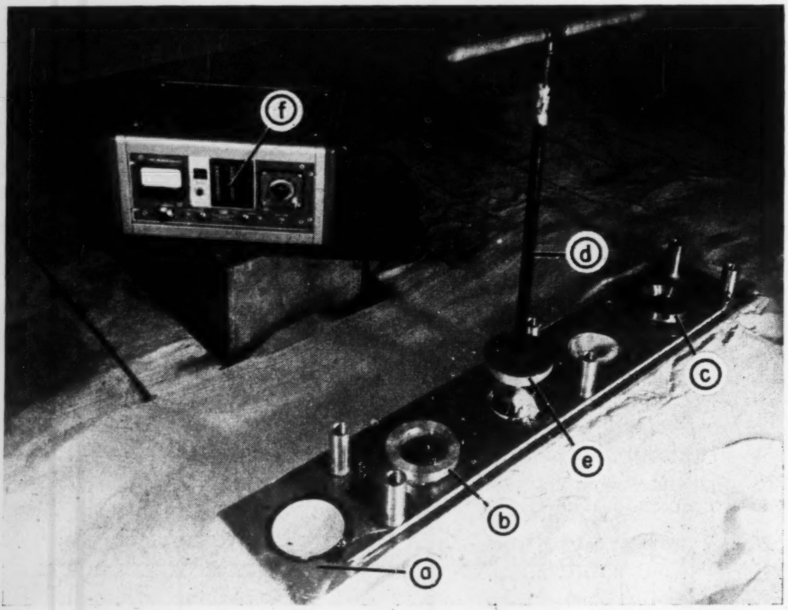
FIG. 2.—Set-up for 3-ft Source-Detector Distance. Source and Detector Lowered below Soil Surface.

rather closely; in the field it is more difficult to control this distance accurately enough. To overcome this difficulty the instrumentation had to be improved.

The general set-up is indicated in Figs. 1 and 2. It consists mainly of an aluminum frame equipped with a winding mechanism to lower radiation source and detector simultaneously to the required depth below the surface.

Figure 3 demonstrates the drilling of holes for the access tubes through openings in the bottom plate. These openings kept the access tubes rigidly at either 1, 2, 3, or 4 ft distances.

Details of the radiation source container and detector are shown in Figs. 4, 5, 6, and 7. During transportation the radiation source of 50 millicurie Co^{60} is shielded in all directions by a minimum of 2.50 in. lead. A lockable bar prevents any unauthorized removal of the radiation source. Only the scintillation crystal of the detector unit is equipped with a directional shield acting as collimator. Both radiation source and detector were enclosed in plastic tubes of 3.50 in. outside diameter and could be lowered simultaneously into access tubes of 4.50 in. outside diameter. No attempt was made at this time to reduce these diameters.



a Bottom plate.

b, c Access tubes inserted for 3-ft source-detector distance.

d Auger to drill hole in soil for access tube at 2-ft source-detector distance.

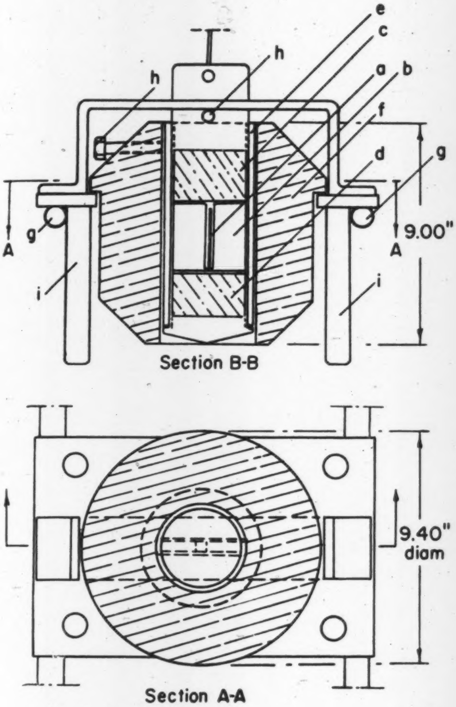
e Centering disk for *d*.

f Decade counter comprising: Electronic register from 1 to 10^3 counts, mechanical register from 10^3 to 10^6 counts and pre-set timer.

FIG. 3.—Set-up to Drill Holes into Soil for Access Tubes at Required Source-Detector Distance.

Correction Factors.—At a distance, x, of 4 ft between radiation source and detector the scaler had to operate at that voltage which produced the largest source to background ratio. This occurred at High Voltage Control Settings (H.V.C.S.) on the scaler of 860 (Fig. 8).

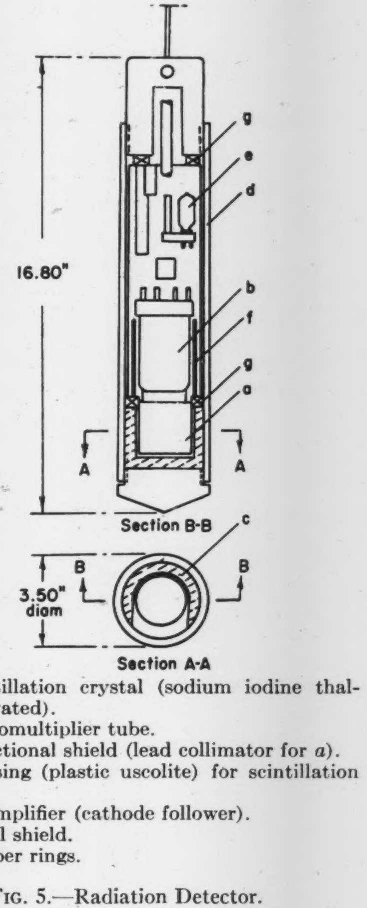
For 3-ft distance, x, the scaler voltage had to be decreased to a H.V.C.S of 600 (Fig. 9). At this lower voltage the radiation counting rate was reduced to slightly below the maximum permissible speed of the mechanical counter on the scaler. However, lowering the voltage required a correction factor. This correction factor applies to readings of x of less than 4 ft to make all counts comparable.



a Irradiated Co^{60} wire.
b Lucite cylinder for *a*.
c, *d* Lead cylinders shielding top and bottom of *a*.
e Housing (plastic uscolite) for *a*, *b*, *c*, and *d*.
f Lead shield for *e*.
g Handle bars to transport.
h Safety locks to hold *e* in *f*.
i Supports for *f*.

FIG. 4.—Radiation Source Container.

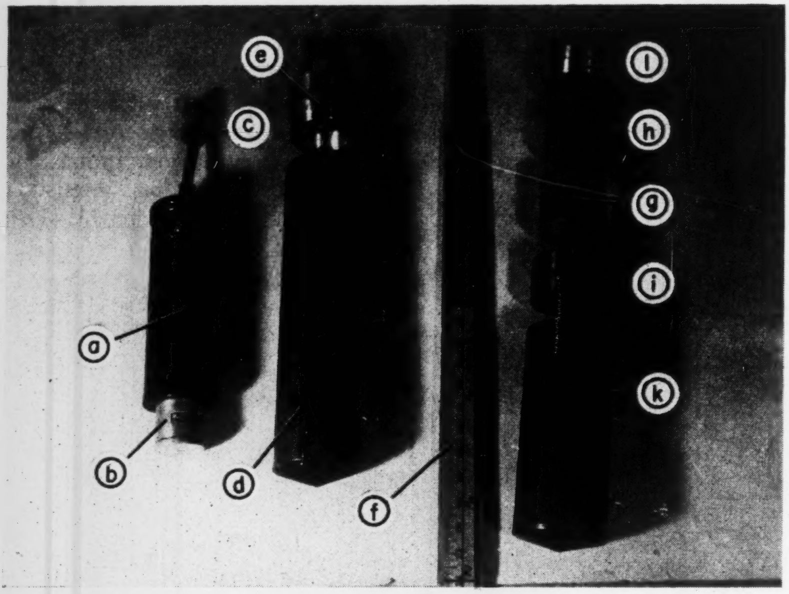
Let the correction factor be defined by the ratio of counts at 860 H.V.C.S for $x = 4$ ft to counts at any lower H.V.C.S. In Fig. 10 are plotted on the X-axis the H.V.C.S and on the Y-axis the corresponding correction factors. At $x = 3$ ft or H.V.C.S = 600, the correction factor becomes 11.17, the best operable range. For values of x less than 3 ft, however, the correction factors increase too much for accurate determination of the transmitted radiation intensity. This difficulty can be avoided by (1) using a scaler with higher permissible counting rate, that is, by adding another electronic decade scaling strip, or (2) reducing the strength of the radiation source for example interposing a lead shield between source and detector.



a Scintillation crystal (sodium iodine thallium activated).
b Photomultiplier tube.
c Directional shield (lead collimator for *a*).
d Housing (plastic uscolite) for scintillation unit.
e Preamplifier (cathode follower).
f Metal shield.
g Rubber rings.

FIG. 5.—Radiation Detector.

A second correction factor is necessary



Detector Parts:

- *a* Steel cylinder containing photomultiplier tube and preamplifier (cathode follower).
- *b* Scintillation crystal (sodium iodide thallium activated).
- *c* Plug for cable connecting I to counter.
- *d* Housing (plastic uscolite) for *a* and *b*.
- *e* Cover (aluminum) for *d*.
- *f* Scale indicating depth below soil surface to be attached to *e*.

Source Parts (Inner Cylinder):

- *g* Lucite cylinder for irradiated Co^{60} wire.
- *h, i* Lead cylinders shielding top and bottom of *g*.
- *k* Housing (plastic uscolite) for *g*, *h*, and *i*.
- *l* Cover (steel) for *k*.

FIG. 6.—Detector and Source Disassembled.

to compensate for the radioactive source decay. The decay extended over the 4-month testing period. This correction factor can be determined as follows: The equation for the basic radiation law is:

$$N = N_o e^{-\lambda t} \qquad (1)$$

where:

N = number of atoms finally present,
N_o = number of atoms initially present,
λ = decay constant, and
t = time of decay.

Let T = half-life time = 5.30 yr for Co^{60},

then $N = \dfrac{N_o}{2}$,

$\dfrac{N_o}{2} = N_o e^{-\lambda t}$ or $2 = e^{+\lambda t}$,

and $\lambda t = \ln 2$ or $\lambda = \dfrac{0.693}{5.3} = 0.1308$.

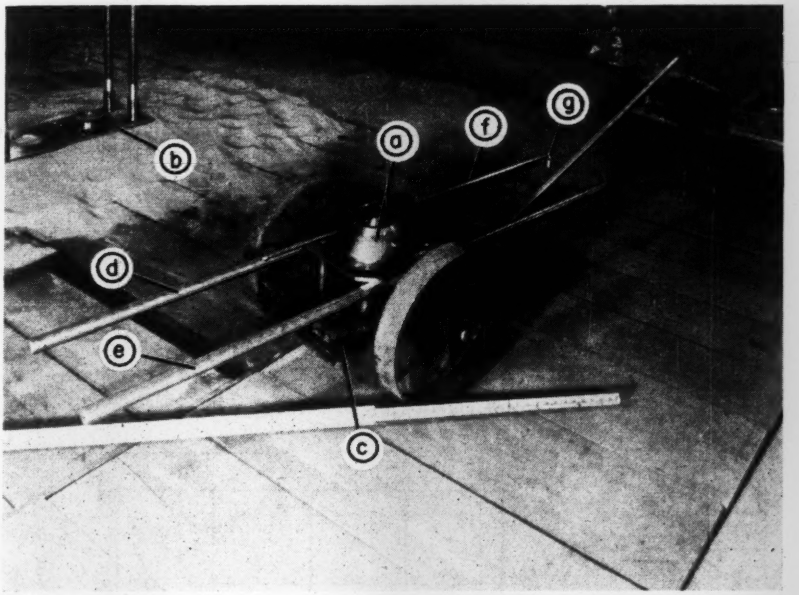
Hence, $F = \dfrac{N_o}{N} = e^{+\lambda t}$, where F = correlation factor.

Finally the monthly corrections due to decay are: after the 1st month = $\dfrac{e^{0.1308}}{12}$ = 1.012, after the 2nd month = $\dfrac{e^{0.1308}}{6}$ =

1.021, after the 3rd month $= \frac{e^{0.1308}}{4} =$ 1.032, and after the 4th month $= \frac{e^{0.1308}}{3}$ $= 1.043$.

Calibration.—In contrast to the laboratory tests, for all field experiments only the sand cone method could be used to calibrate soil densities *versus* counter readings. The probable error of several sand cone density determinations averaged ±2.5 lb per cu ft.

This error was due partly to the inherent inaccuracy of the sand cone method and partly due to the inhomogeneity of the soil in each test bed. This inhomogeneity could not be avoided in spite of careful mixing of the soil before placing it into the various beds (6).



a Isotope container.
b Bottom plate.
c Cart for transportation of *a*.
d, *e* Handle bars for moving *a* from *c* to *b*.
f Bar to lock radiation source in *a*.
g Pad lock preventing disengagement of *f*.

FIG. 7.—Isotope Container on Transport Cart.

Regardless of the source of these deviations, the sand cone method limits the accuracy of any comparison with radiation methods.

In Fig. 11 some of the experimental values obtained from field investigations are summarized. Again on the X-axis the wet soil density, ρ, in lb per cu ft is plotted, using a linear scale. On the Y-axis the measured radiation intensity, I, in cpm, as transmitted through 13 types of soils, has been indicated, using a logarithmic scale. All counter readings are multiplied with the corresponding correction factors and the background level has been subtracted.

Source detector distances, x, of 3 and 4 ft were selected. The geometrical setup remained identical for all field measurements.

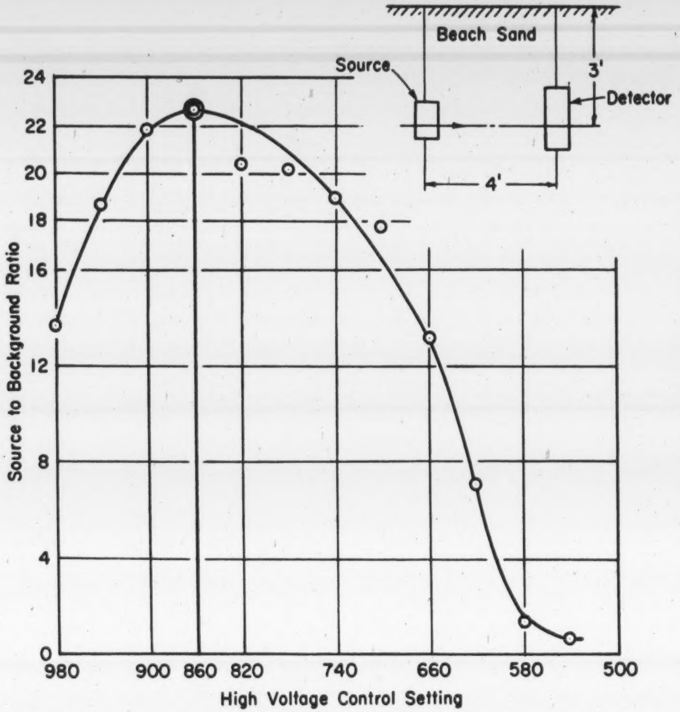


FIG. 8.—Source to Background Ratios *versus* High-Voltage Control ($x = 4$ ft).

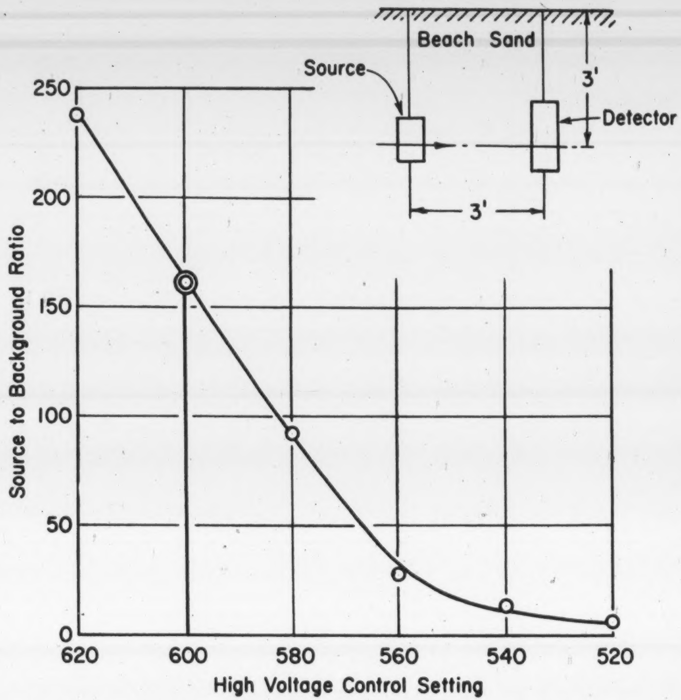


FIG. 9.—Source to Background Ratio *versus* High-Voltage Control ($x = 3$ ft).

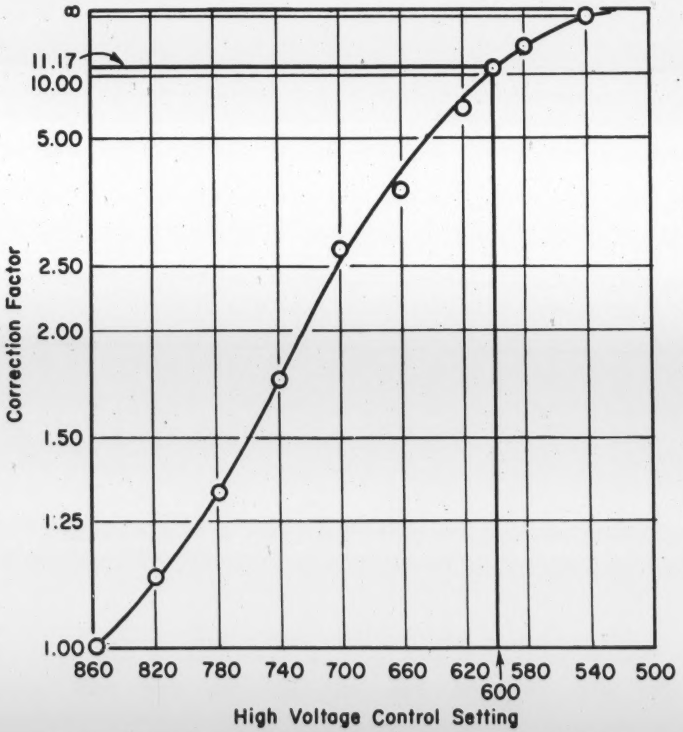


FIG. 10.—Correction Factor *versus* High-Voltage Control Setting.

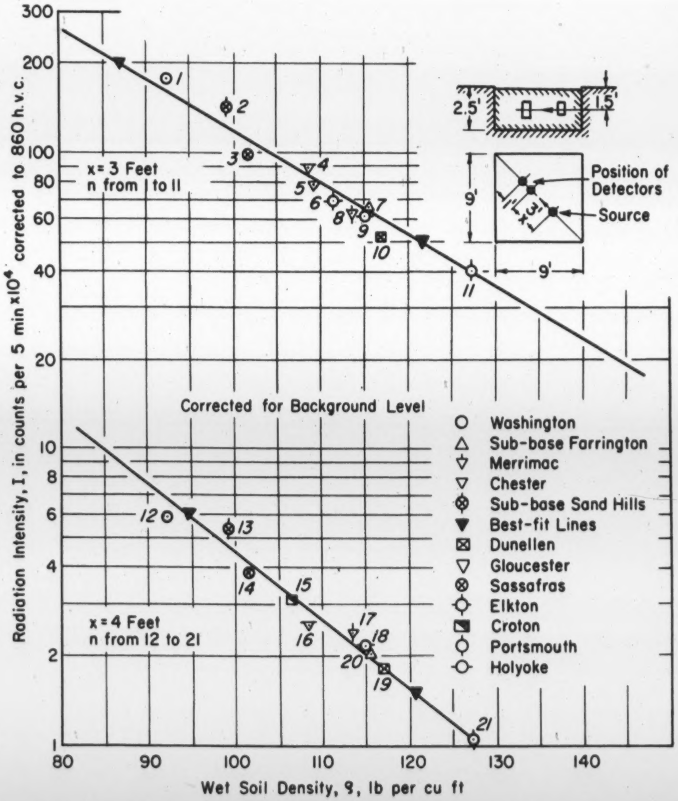


FIG. 11.—Transmitted Radiation Intensity *verus* Wet Soil Density for Field Experiments.

Scattering was considered small enough to use equations for the primary beam only (4).

STATISTICAL ANALYSIS (LEAST SQUARE METHOD)

The general equation applied for the statistical analysis is:

$$\Sigma \ln I = \Sigma a_1 + \Sigma a_2 \ln x + \Sigma a_3 \rho x \quad ..(2)$$

which can be written in the form:

$$\Sigma \ln I \ln x = \Sigma a_1 \ln x + \Sigma a_2 (\ln x)^2 + \Sigma a_3 \ln x(\rho x)$$

or

$$\Sigma \ln I(\rho x) = \Sigma a_1 \rho x + \Sigma a_2 \rho x \ln x + \Sigma a_3 (\rho x)^2 \quad ..(3)$$

where the three variables are:

ρ = wet soil density in lb per cu ft,

x = distance between radiation source and radiation detector in feet, and

I = transmitted radiation energy through the soil in cpm, and where the three unknown parameters are:

a_1 = composite constant depending mainly upon initial intensity of radiation source and a specific distance between source and detector,

a_2 = exponent taking care of the reduction of transmitted radiation energy with approximately the square of the variable distance, x, and

a_3 = constant depending upon the absorption coefficient of the transmitting medium.

The purpose of the statistical analysis is the determination of the parameters a_1, a_2 and a_3. The physical concepts which led to the derivation of these equations are described elsewhere (3, 4).

Parameters a_1, a_2 *and* a_3.—Solving these equations for a_1, a_2, a_3 by determinants yields:

$$a_1 = \frac{\begin{vmatrix} \Sigma \ln I & \Sigma \ln x & \Sigma \rho x \\ \Sigma \ln I \ln x & \Sigma (\ln x)^2 & \Sigma \ln x(\rho x) \\ \Sigma \ln I\,(\rho x) & \Sigma \rho x \ln x & \Sigma (\rho x)^2 \end{vmatrix}}{\begin{vmatrix} \mathrm{n} & \Sigma \ln x & \Sigma \rho x \\ \Sigma \ln x & \Sigma (\ln x)^2 & \Sigma \ln x(\rho x) \\ \Sigma (\rho x) & \Sigma \rho x \ln x & \Sigma (\rho x)^2 \end{vmatrix}} = \frac{\Delta_1}{\Delta} \quad \ldots\ldots\ldots\ldots\ldots (4)$$

or

$$a_2 = \frac{\begin{vmatrix} \mathrm{n} & \Sigma \ln I & \Sigma \rho x \\ \Sigma \ln x & \Sigma (\ln x) \ln I & \Sigma \rho x \ln x \\ \Sigma (\rho x) & \Sigma \rho x \ln I & \Sigma (\rho x)^2 \end{vmatrix}}{\Delta} = \frac{\Delta_2}{\Delta} \quad \ldots\ldots\ldots\ldots\ldots (5)$$

and

$$a_3 = \frac{\begin{vmatrix} \mathrm{n} & \Sigma \ln x & \Sigma \ln I \\ \Sigma \ln x & \Sigma (\ln x)^2 & \Sigma \ln I \ln x \\ \Sigma \rho x & \Sigma \rho x \ln x & \Sigma \ln I\,(\rho x) \end{vmatrix}}{\Delta} = \frac{\Delta_3}{\Delta} \quad \ldots\ldots\ldots\ldots\ldots (6)$$

The values based on experimentally obtained data for I (Fig. 1) and referring to the distance x for 3 and 4 ft are used in the numerical solution of Eq 3.

By substituting the Σ values to determine denominator Δ, numerators Δ_1, Δ_2, and Δ_3 into Eqs 4, 5, and 6 follows:

$$\Delta = \begin{vmatrix} 21 & 25.9476 & 8{,}016.2 \\ 25.9476 & 32.4939 & 10{,}067.9893 \\ 8{,}016.2 & 10{,}067.9893 & 3{,}144{,}409.3 \end{vmatrix} = +2.08716 \times 10^5$$

$$\Delta_1 = \begin{vmatrix} 251.10907 & 25.9476 & 8{,}016.2 \\ 305.19921 & 32.4939 & 10{,}067.9893 \\ 93{,}636.98 & 10{,}067.9893 & 3{,}144{,}409.3 \end{vmatrix} = +5.28202 \times 10^6$$

$$\Delta_2 = \begin{vmatrix} 21 & 251.10907 & 8{,}016.2 \\ 25.9476 & 305.19921 & 10{,}067.9893 \\ 8{,}016.2 & 93{,}636.98 & 3{,}144{,}409.3 \end{vmatrix} = -1.39387 \times 10^6$$

$$\Delta_3 = \begin{vmatrix} 21 & 25.9476 & 251.0907 \\ 25.9476 & 32.4939 & 305.19921 \\ 8{,}016.2 & 10{,}067.9893 & 93{,}636.98 \end{vmatrix} = -2.80136 \times 10^3$$

or

$$\hat{a}_1 = \frac{\Delta_1}{\Delta} = +\frac{5.28202 \times 10^6}{2.08716 \times 10^5} = +25.30721$$

$$\hat{a}_2 = \frac{\Delta_2}{\Delta} = -\frac{1.39387 \times 10^6}{2.08716 \times 10^5} = -6.67831$$

$$\hat{a}_3 = \frac{\Delta_3}{\Delta} = -\frac{2.80136 \times 10^3}{2.08716 \times 10^5} = -1.34220 \times 10^{-2}$$

Thus, the equation for the transmitted intensity becomes from Eq 2:

$$\ln I = 25.30721 - 6.67831 \ln x - 0.134220 \rho x$$

or solving for ρ

$$\rho = \frac{1888.6}{x} - 74.505 \frac{\ln I}{x} - 497.56 \frac{\ln x}{x} \quad \text{.. (7)}$$

The parameter $a_3 \pm 0.0134$ ft^2 per lb, represents the experimentally obtained mass absorption coefficient. This value for a_3 lies between the theoretical μ_p (4) for the primary beam only and the theoretical μ_{p+s} for the primary beam and the scattered photons. Hence:

$$\mu_{p+s} < a_3 < \mu_p$$

or

$$0.00636 < 0.0134 < 0.025$$

It must be kept in mind that the source used in the field experiments was not collimated. Therefore, the experimental $\mu = a_3$ did not approach the theoretical upper limit of μ_p.

"Best-Fit Values" (x = 3 ft and 4 ft):

Arbitrarily selected values of I for x equal 3 and 4 ft are evaluated. From each set of values the soil density, ρ, can be determined using Eq 7.

The finally obtained values for ρ are connected by straight lines in Fig. 11.

Standard Error of Estimate:

The standard error of estimate, σ_{se}, can be determined from the following equation (1):

$$\sigma_{se} = \sqrt{\frac{1}{n-2} \sum_{i=1}^{n} \left(\ln I_i - \sum_{p=1}^{3} a_p u_{p-1} \right)^2} \quad \text{.. (8)}$$

where:

n = total number of observations,
i = observation number, and
p = parameter number.

Equation 8 yields for $n = 21$:

$$\sigma_{se} = 0.0989$$

This standard error of estimate is low

when compared with $\sigma_{se} = 0.1159$ gained from previous laboratory experiments and $\sigma_{se} = 0.251$ obtained in earlier field tests (4).

Both the preceding laboratory and earlier field investigations were made without the improved technique described above.

From $\sigma_{se} = 0.0989$ follows that the transmitted radiation intensity, I, varies between the limits:

$$\hat{I}e^{-0.0989} \leqslant I \leqslant \hat{I}e^{+0.0989}$$

or

$$0.906\hat{I} \leqslant I \leqslant 1.104\hat{I},$$

where $\hat{I}$ represents the so-called "best fit" for I.

The standard error of estimate means that about 68 per cent of all experimental points will fall within the above limits. At $x = 3$ ft the deviation in terms of ρ would be approximately ±2.3 per cent.

Selecting an average soil density of 100 lb per cu ft, a deviation of ±2.3 per cent is equal to ±2.3 lb per cu ft. Keeping in mind that the sand cone method yielded a probable error of the same magnitude, it seems reasonable to assume that the radiation method is apparently the more accurate of the two.

Results:

For the soils investigated so far Eq 7 represents an adequate solution of the correlation between wet soil density and measured radiation intensity regardless of the type of soil acting as transmitting medium.

Errors in the horizontal distance or differences in the depth below soil surface of source and detector cause faulty results. The improved technique has reduced these errors as can be deducted from the decreased standard error of estimate.

Larger source detector distances integrate the soil density regardless of inclusion of insignificant soft or hard spots (stones). Furthermore, larger values of x avoid the pitfalls of spot checks inherent, for example, to the sand cone method. Finally, larger distances require less observation points.

Shorter source detector distances, however, may become necessary in case small contact areas (machine foundations) are sensitive to localized soft spots.

It must be clearly understood that so far the radiation method represents rather a supplement to conventional methods than a foolproof substitute.

Investigations are under way studying radiation methods to determine *dry* soil density directly. Pilot tests combining a Ra-Be source emitting γ-rays and neutrons and a lithium iodide (europium activated) crystal detector with and without cadmium shield showed promising results.

CONCLUSIONS

1. Statistical analyses become almost indispensable for investigation in the realm of isotopic disintegration processes, in particular for cases where random values predominate such as measurements of radiation energy transmitted through soils.

2. When determining the three parameters inherent to equations governing the relationship between soil density, transmitted radiation intensity, and distance between radiation source and detector, the third parameter representing the absorption coefficient is of particular importance.

3. The nonavoidable inaccuracy of the standard sand cone method imposes certain limitations to any comparison with radiation methods.

4. Further analyses referring to the effect of primary beam and scattering, and investigation to correlate laboratory and field experiments are necessary.

5. The Eq 2 is not completely justified from the physical point of view, due to the assumption that the measured

radiation intensity I is the random variable and the soil density ρ, a variable taken at fixed values.

Further investigations might indicate that the reverse will yield more reliable values, that is, in combination with improved radiation counters, I, will become a variable taken at fixed values and ρ, the random variable.

Acknowledgment:

All experiments were carried out at the Non-Destructive Testing Laboratories of the College of Engineering at Rutgers University.

The authors would like to express their sincerest appreciation to the Research Council of Rutgers University, who supplied the funds for this project.

Furthermore, the writers are greatly indebted to Dr. E. C. Easton, Dean of the College of Engineering, J. J. Slade, Jr., Research Director of the Bureau of Engineering Research, for their continued interest and valuable suggestions, and L. F. Nanni, Professor of Engineering Mechanics, for his assistance with the statistical analysis.

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(3) R. K. Bernhard and M. Chasek, "Soil Density Determination by Means of Radioactive Isotopes," *Non-destructive Testing*, Vol. XI, No. 8, Nov.–Dec., 1953, pp. 17–23; Vol. XII, No. 1, Jan.–Feb., 1954, p. 40.

(4) R. K. Bernhard and M. Chasek, "Soil Density Determination by Direct Transmission of Gamma Rays," *Proceedings*, Am. Soc. Testing Mats., Vol. 55, p. 1199 (1955).

(5) I. Goldberg and L. J. Trescony, "Measurement of Moisture Content and Density of Soil Masses Using Radioactive Methods." Paper presented at Pacific Coast Regional Conference on Clays and Clay Technology, June 25–26, 1954.

(6) F. C. Rogers and H. C. Nicola, "Frost Action Studies of Thirty Soils in New Jersey." Symposium on Frost Heave and Frost Action in Soil, Highway Research Board. (Issued as special publication, 1951.)

DISCUSSION

Mr. C. H. Scholer.[1]—Is the ordinary laboratory technician allowed to use this kind of material or must the work be done through some authorized radiation laboratory when dangerous radioactive materials are involved?

Mr. R. K. Bernhard (*author*).—Before starting experiments with radioactive material, authorization from the U. S. Atomic Energy Commission has to be obtained. Similar to X-ray inspection methods where radiation and additional high-voltage hazards exist, the operator has to be familiar with the required safety precautions.

Mr. M. D. Morris.[2]—The authors compare the gamma ray method with other density methods. Were any results obtained by (and if so, were they in any way comparable to) a balloon density method. Nothing was mentioned of this, only sand cone correlations.

Mr. Bernhard.—Only comparisons with the standard sand cone density method have been made so far.

Mr. Morris.—I do not know myself, but there will soon be a paper to compare the methods. I would be interested to hear first what that has to say.

Mr. Robert Horonjeff.[3]—How near the surface is the equipment reliable? Could measurements be made about 4 in. below the surface?

Mr. Bernhard.—No experiments have been made with the direct transmission methods at depths of less than 6 in. below the soil surface. It is our understanding that instruments have been developed which can be placed on the surface and reach a depth of about 6 in. These instruments are based however on the back reflection principle.

(*Authors' closure*).—Methods ought to be found for wet and dry soil density determination which are more accurate and faster than the procedures known so far. Whether radiation or other principles will prevail remains to be seen. Committee D-18 on Soils should encourage all research in this direction. The authors would be highly gratified if their studies have created enough interest to foster further investigations in this line.

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[1] Professor of Applied Mechanics, Kansas State College, Manhattan, Kan.

[2] Eastern Representative, Soiltest Inc., New York, N. Y.

[3] Institute of Transportation and Traffic Engineering, University of California.

EVALUATION OF LIME FOR STABILIZATION OF LOESS*

By J. G. Laguros,[1] D. T. Davidson,[1] R. L. Handy,[1] and T. Y. Chu[1]

Synopsis

In lime stabilization of soils it is recognized that there are wide variations between different soils, and treatments are adjusted accordingly. In view of this complication it has been reassuring to the soil engineer that he could look on lime as lime and not have to worry about it. Now, research shows that some limes are more effective than others, and data in this paper emphasize the need for testing and evaluating a lime to determine its optimum use. Test results with lime-stabilized loess are related to calcium:magnesium ratio in the lime and to the use of quicklime *versus* hydrated lime. On the basis of compressive strength, quicklime appears to be more effective with loess than the equivalent amount of hydrated lime, and dolomitic lime is more effective than calcitic lime. The greater effectiveness of dolomitic lime is also indicated in artificial lime mixtures. Data are given comparing the California Bearing Ratio (CBR), plasticity indices, and shrinkage limits obtained after treatment with hydrated lime and quicklime. A high-temperature curing study is also included.

In Iowa and adjacent states, loess of Wisconsin geologic age is a major surficial material, and a method for treating loess to make it suitable for road base course construction would be highly desirable. With this objective in mind, research has recently been conducted in Iowa with lime. It is interesting to note that one of the first field tests of lime stabilization was a lime-loess road constructed in the 1920's (**7**)[2] in southwest Iowa. Partly due to the lack of a wearing surface, that road failed. The laboratory work described in this paper extends over the past several years and re-examines the feasibility of lime-loess construction in southwest Iowa. Samples of loess were carefully chosen to represent the friable to plastic range, and tests were conducted using a number of varieties of lime. In the comparisons, some interesting differences were found, particularly with reference to the lime, and contrasts could be made between quicklime and hydrated lime and between calcitic and dolomitic lime. The purpose of this paper is thus twofold: to resolve the question of the suitability of lime for loess stabilization and to point up the variable effectiveness of different limes.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] Instructor of Civil Engineering, Professor of Civil Engineering, Research Geologist of the Engineering Experiment Station, Iowa State College, Ames, Iowa; and Associate Professor of Civil Engineering, New York University, New York, respectively.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1315.

TABLE I.—BRIEF DESCRIPTION OF LOESS SAMPLES.

Property	Sample 20-2 (IV)	Sample 44A-1
Location	Harrison Co.	Fremont Co.
Geologic Material	Friable Loess	Plastic Loess, Leached
Thickness	Over 100 ft	15 to 20 ft
Soil Series	Hamburg	Marshall
Horizon	C	C
Engineering Classification (Am. Assn. State-Highway Officials)[a]	A-4 (8)	A-7-6 (18)

[a] Standard Specifications for Materials for Embankments and Subgrades (M 57–55), 1955 Standard Spec. for Highway Mats., Part I, p. 54.

TABLE II.—PROPERTIES OF LOESS SAMPLES.

Property		Sample 20-2(IV)	Sample 44A-1
Textural Composition,[a] per cent by weight	Gravel	0	0
	Sand	0.7	0.2
	Silt	78.3	58.0
	Clay	21.0	41.8
	Colloidal	15.8	31.0
Textural Classification[b]		Silty Clay Loam	Silty Clay
Physical Properties[c]	L.L., per cent	32.9	53.1
	P.L., per cent	21.1	25.7
	P.I., per cent	11.7	27.4
	S.L., per cent	28.3	19.9
	C.M.E., per cent	15.2	ND
	F.M.E., per cent	26.4	ND
	sp gr[c] 25 to 4 C	2.68	ND
Predominant Clay Mineral		Nontronite Illite Interlayer	Nontronite Illite Interlayer
Predominant Exchangeable Cations		Calcium	Calcium
Chemical Properties	Cation exchange capacity milliequivalent per 100 g	13.4	28.2
	pH[d]	7.8	6.2
	Carbonates,[e] per cent	10.2	ND
	Organic Matter,[e] per cent	0.2	ND

ND—Not determined.

[a] Gravel—above 2.0 mm, sand—2.0 to 0.074 mm, silt—0.074 to 0.005 mm, clay—below 0.005 mm, colloidal—below 0.001 mm.

[b] Textural classification is based on the U. S. Bureau of Public Roads system except that 0.074 mm was used as the lower limit of the sand fraction.

[c] L.L. = Liquid limit.
P.L. = Plastic limit.
P.I. = Plasticity index.
S.L. = Shrinkage limit.
C.M.E. = Centrifuge moisture equivalent.
F.M.E. = Field moisture equivalent.

[d] Determined on the fraction passing No. 10 sieve.

[e] Determined on the fraction passing No. 40 sieve.

TABLE III.—PROPERTIES OF HYDRATED LIME USED.

Property		Sample and Type of Lime					
		H-1 Calcitic	H-2 Calcitic	H-3 Dolomitic Dihydrate	H-4 Calcitic	H-5 Calcitic	H-6 Dolomitic Monohydrate
Fineness, per cent	Material passing No. 200 sieve	ND[a]	99.50	ND	99.4	99.0	99.55
	Material passing No. 325 sieve	99.0	98.75	ND	ND	98.4	99.24
Chemical analysis, per cent	Silicon dioxide	0.80	0.25	0.57	0.52	0.58	0.6
	Iron and aluminum oxide	0.82	0.26	1.06	0.18	0.44	1.1
	Magnesium oxide	0.49	0.17	29.72	0.59	0.49	33.2
	Calcium carbonate	0.77	ND	ND	1.41	1.50	ND
	Total calcium hydroxide	97.82	ND	ND	ND	96.46	ND
	Available calcium hydroxide	97.38	94.85	ND	94.58	ND	ND
	Loss on ignition	24.56	23.15	27.06	24.52	ND	ND
	Total calcium figured as CaO	ND	74.53	ND	ND	ND	ND
	Calcium oxide	ND	ND	42.21	74.21	73.00	48.3
	Combined H_2O	ND	22.83	ND	ND	0.45	16.8
	Carbon dioxide	ND	ND	2.90	0.62	ND	ND
	Sulfur trioxide	ND	ND	0.105	ND	ND	ND
	Calcium magnesium ratio	181:1	512:1	1.7:1	146:1	180:1	1.7:1

[a] Not determined.

TABLE IV.—PROPERTIES OF QUICKLIME USED.

Property		Sample and Type of Lime					
		Q-1 Calcitic	Q-2 Calcitic	Q-3 Dolomitic	Q-4 Calcitic	Q-5 Calcitic	Q-6 Dolomitic
Fineness, per cent	Material passing No. 50 sieve	100.0	100.0	ND	ND	ND	99.98
	Material passing No. 200 sieve	ND[a]	78.31	ND	ND	ND	97.01
	Material passing No. 325 sieve	ND	71.15	ND	ND	99.0	ND
Chemical analysis, per cent	Silicon dioxide	1.97	0.34	0.67	0.38	0.75	0.7
	Iron and aluminum oxide	1.17	0.30	1.00	0.28	0.53	1.4
	Magnesium oxide	0.84	0.14	40.41	0.68	0.65	39.8
	Calcium carbonate	7.94	ND	ND	ND	2.76	ND
	Total calcium oxide	96.02	ND	56.59	96.45	95.00	58.1
	Available calcium oxide	92.52	93.80	ND	94.20	ND	ND
	Loss on ignition	4.45	0.66	1.70	2.03	ND	ND
	Total calcium figured as CaO	ND	97.75	ND	ND	ND	ND
	Total sulfur	ND	ND	0.037	ND	ND	ND
	Carbon dioxide	ND	ND	ND	1.27	ND	ND
	Calcium sulfate	ND	ND	ND	ND	0.10	ND
	Calcium : magnesium ratio	132:1	837:1	1.67:1	164:1	174:1	1.7:1

[a] Not determined.

The mechanism of lime stabilization of loess is also discussed.

MATERIALS

Properties of Loess:

In Iowa, the loess varies texturally from silty loam to silty clay. Clay mineral compositions qualitatively are believed to be the same (6), the principal clay mineral being an interlayer mixture of montmorillonite and illite. Cation exchange capacity is approximately proportional to the clay content.

The thick loess occurring near river valley and till plain source areas is characteristically calcareous and low in clay. Farther away from the sources the loess deposits are thinner, leached of carbonates, and higher in clay.

Two samples of C-horizon loess were used in the lime stabilization studies; sample No. 20-2(IV) was selected to represent the friable, calcareous loess, and sample No. 44A-1 to represent the plastic, leached loess. Properties of the two samples, which for simplicity will be referred to as the friable loess and the plastic loess, are given in Tables I and II.

Properties of Lime:

Different commercial limes were furnished by the National Lime Assn and include calcitic and dolomitic hydrated limes and quicklimes which meet the various standards of the Association and of ASTM (7). Properties of the hydrated limes are given in Table III and properties of the quicklimes in Table IV. Hydrated dolomitic limes are of the type N ("Monohydrate") in which magnesium occurs as the oxide and of type S ("Dihydrate") in which magnesium occurs as the hydroxide.

OPTIMUM MOISTURE CONTENT

Prior to the main testing program, the optimum moisture content for maximum density was compared to the optimum moisture content for maximum strength by plotting the strength-density and the moisture-density relationships and reading the highest points. Specimens were molded with 6 per cent hydrated lime H-1 and with the approximate equivalent of quicklime Q-1, 4.5 per cent. The difference between optimum moisture contents for compaction and for strength was 2 or 3 per cent in the case of the hydrated lime, the higher moisture content giving lower density and highest strength. However, the difference in strength at the two moisture contents is slight: of the order of 4 to 8 per cent. With quicklime the differences are even less, and for convenience the optimum moisture content for maximum density was used in the subsequent testing program.

COMPRESSIVE STRENGTH

Method:

Each lime was added to the soil dry, the batch mixed, and water added. Specimens 2 in. in diameter and 2 in. high ($h/d = 1$) were then molded to a density of not less than 95 per cent of standard Proctor density (ASTM Method D 698[3]). A drop hammer molding apparatus was used; three specimens were molded for each combination of lime and loess. Specimens were cured for 7 days in a cabinet maintained at 70 ± 3 F and a relative humidity of not less than 90 per cent. The specimens were then soaked in distilled water for 24 hr prior to testing; compressive strengths were determined using a load applied at 0.10 in. per min.

Test Results:

The change in compressive strength of lime-stabilized loess with various percentages of different limes is shown

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[3] Tentative Method of Test for Moisture-Density Relations of Soils (D 698 – 42 T), 1955 Book of ASTM Standards, Part 3, p. 1789.

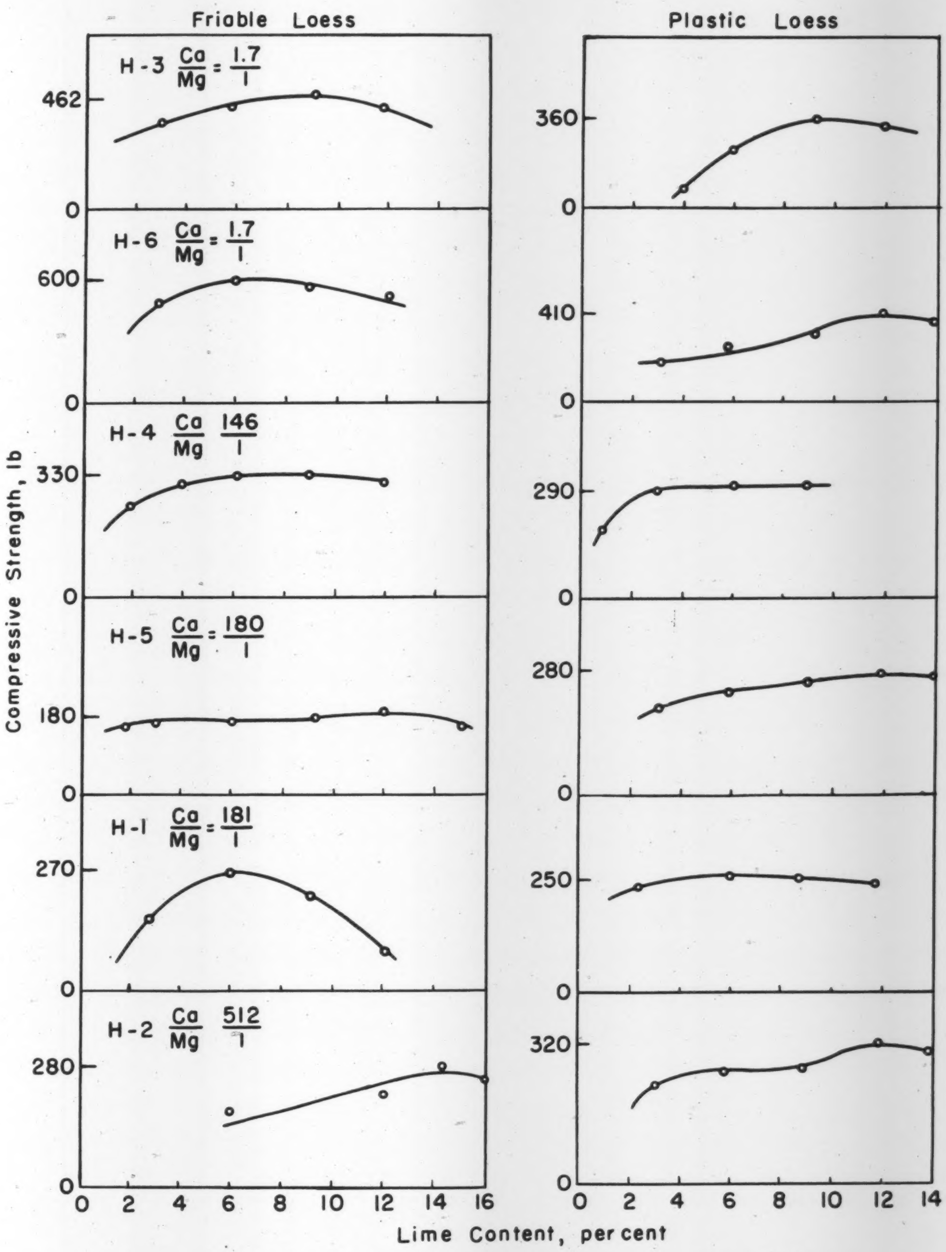

FIG. 1.—Effect of Hydrated Lime Content on the Compressive Strength of Loess.

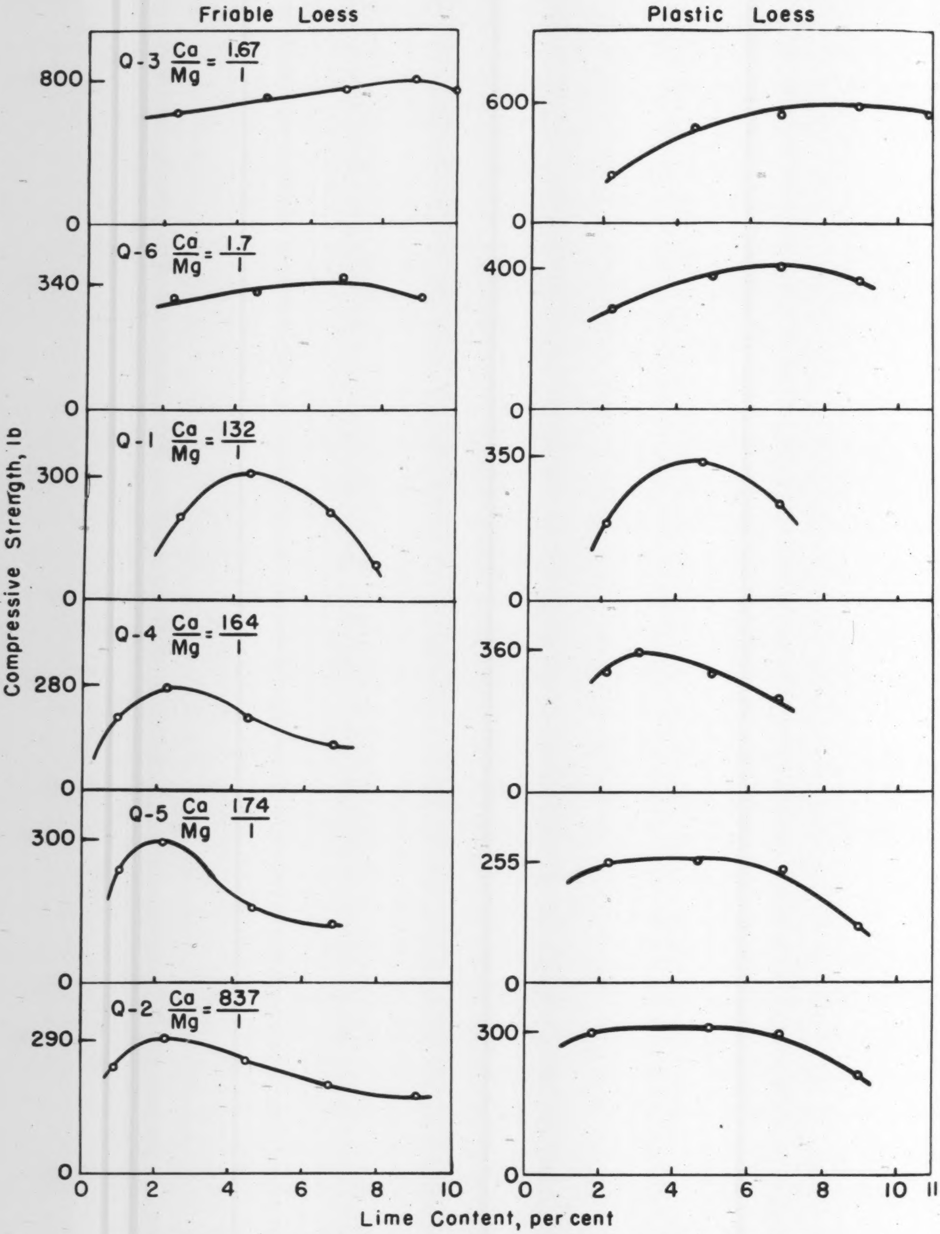

FIG. 2.—Effect of Quicklime Content on the Compressive Strength of Loess.

graphically in Figs. 1 and 2. All specimens with zero per cent lime failed on immersion in water. It can be seen that there is a considerable difference in the shape of the curves. Quicklimes show a decided optimum content which varies from 2.5 to 9 per cent lime. The curve peaks tend to be less pronounced in the case of the plastic loess. Some hydrated limes show compressive strength peaks; some do not. Curves which do not give decided peaks usually reach their strength quickly and then show a flattening out above a lime content of about 3 per cent.

TABLE V.—RELATION OF IMMERSED COMPRESSIVE STRENGTHS OF LIME-STABILIZED LOESS TO PER CENT AND KIND OF LIME.

Properties of Lime			Results with Friable Loess				Results with Plastic Loess			
Sample	Calcium: Magnesium Ion Ratio	Weight Equiv. to 1 g $Ca(OH)_2$	7-day Compressive Strength, lb			Optimum per cent Lime	7-day Compressive Strength, lb			Optimum per cent Lime
			3 per cent (equiv.) Lime[a]	6 per cent (equiv.) Lime[a]	Optimum per cent Lime		3 per cent (equiv.) Lime[a]	6 per cent (equiv.) Lime[a]	Optimum per cent Lime	
Hydrated Lime										
H-3	1.7	0.905	240	265	462	9	35	275	358	9
H-6	1.7	0.801	425	580	600	6	140	220	413	12
H-4	146	1.01	200	207	335	6	208	210	290	6
H-5	180	1.03	170	170	177	12	195	240	283	12
H-1	181	1.01	210	270	270	6	240	250	250	6
H-2	512	1.01	155	165	268	15	230	265	320	12
Quicklime										
Q-3	1.67	0.664	620	695	800	9	150	485	660	9
Q-6	1.7	0.671	295	300	344	6.5	265	355	400	7
Q-1	132	0.779	260	300	300	4.5	290	360	360	4.5
Q-4	164	0.779	280	195	280	2.5	305	305	358	3
Q-5	174	0.790	295	145	290	2.3	255	250	255	3
Q-2	837	0.772	290	245	294	2.5	305	310	310	4.5

[a] Equivalent on an ionic basis to 3 or 6 per cent $Ca(OH)_2$.

Chemically Equivalent Percentages of Various Limes:

In order to evaluate the different limes on a more or less equivalent basis, amounts were calculated which could give the same number of calcium plus magnesium ions. Allowances were made both for hydration and for difference in atomic weights. For example, 1.0 g of CaO (molecular weight 56.08) is equivalent to 1.32 g $Ca(OH)_2$ (molecular weight 74.11). In a dolomitic lime, 1.0 g of MgO (molecular weight 40.32) is equivalent to 1.84 g of $Ca(OH)_2$. Hydrated dolomitic lime is assumed to be made up of $Ca(OH)_2$ and MgO. Results expressed on an equivalent lime basis are presented in Table V. Strengths were interpolated from the lime content - strength curves.

DISCUSSION

The curves in Figs. 1 and 2 illustrate one important fact: it is possible to add too much lime. Probably the excess lime acts as a filler separating the mineral grains, since hydrated lime crystals have a weak claylike (gibbsite) structure. Probably with continued curing, the excess lime would be utilized, adding to strength. Characteristics of individual curves are probably related to individualities of each lime.

The data in Table V show that quicklime is in general more effective than hydrated lime. With equivalent amounts the average strength is about 40 per cent higher with quicklime. However, exceptions were found. The difference is not believed due to the heat of hydration of the quicklime, since the heat is rapidly dissipated during mixing and immediately after molding. Further work is under way to check this.

Another trend revealed in Table V is for dolomitic limes to give higher strengths than calcitic limes. At equivalent percentages, the effect is not so noticeable, but the strengths at higher percentages are quite different. Dolomitic limes can be used in higher percentages before there is a decrease in strength. Of the hydrated dolomitic limes, the monohydrate (type N) gives higher strength than the dihydrate (type S). However, the different brands of dolomitic quicklime also give a variation in strength, and strengths with one dolomitic quicklime are double the strengths with the other. No explanation for this was found in hydration treatment or calcium:magnesium ratio.

The above discussion points out benefits to be derived from testing different limes prior to selection of one for a stabilized-soil construction. With some combinations the same strength can be realized with one-half or one-third as much lime. This sensitivity of soil to variables in lime should not be entirely unexpected; soils are chemically active and complex. A rational analysis may eventually become possible, but a major obstacle is the lack of adequate data on lime. Particle size and surface chemical factors related to curing are two quantities that need expression. When these quantities become measureable and measured, more definite correlations may be found and applied in the quality control of lime. Until then, laboratory tests on soil-lime mixes seem the only answer.

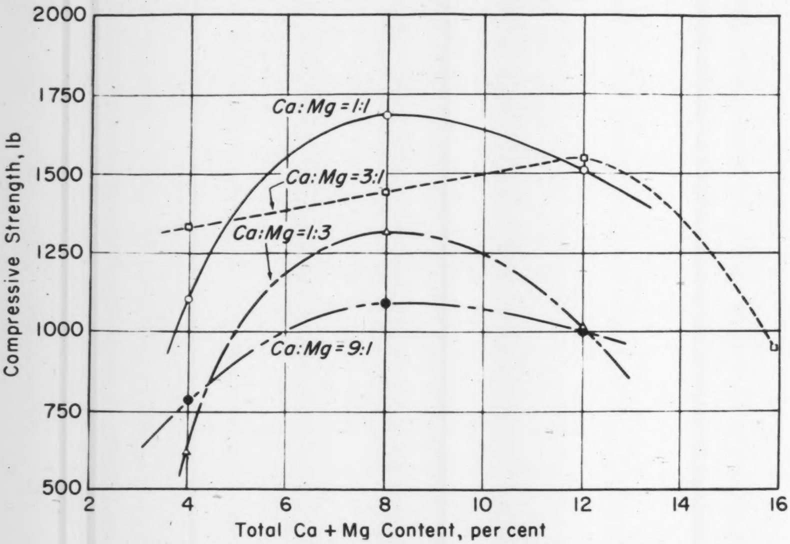

FIG. 3.—Effect of Ca: Mg Ratio on Lime Stabilization of Friable Loess.

With reference to differences in soils, the data in Table V show higher strengths with the friable loess. This is not exactly as expected, since lime is supposed to react best with clays. The significance of this is discussed later under mechanism of lime stabilization.

ARTIFICAL LIME MIXTURES

In order to gain further evidence for some of the relationships discussed above, artificial dolomitic limes were prepared by mixing purified grade $Ca(OH)_2$ and MgO in various calcium to magnesium ratios from 1:9 to 9:1. Results are plotted in Figs. 3 and 4. With both the friable and the plastic loess the ideal lime composition appears to be between 1:1 and 3:1 calcium to magnesium. Strengths dropped when MgO was increased beyond a ratio of 1:1.

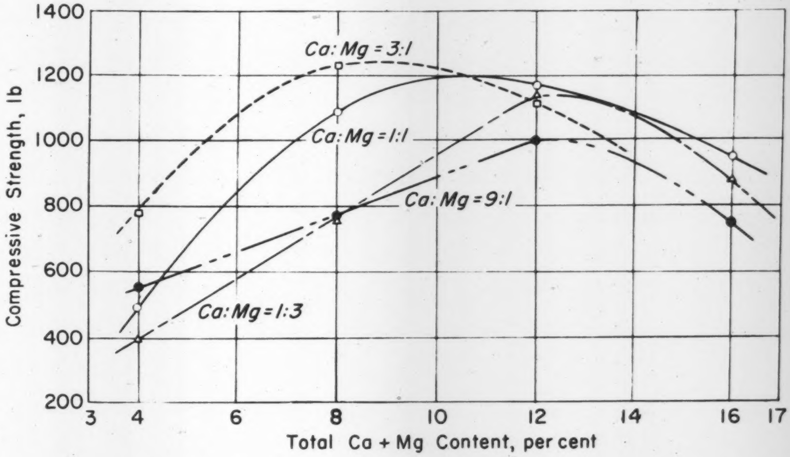

FIG. 4.—Effect of Ca: Mg Ratio on Lime Stabilization of Plastic Loess.

CURING STUDIES

Among the different variables affecting the strength of lime-stabilized loess, curing is of major importance. Its effect on strength is a function of time, temperature, and relative humidity. To study the effect of these factors, curing studies were conducted on loess samples stabilized with limes H-1 and Q-1. Specimens 2 in. high by 2 in. diam were molded, cured, immersed in water for 24 hr, and tested for unconfined compressive strength. Various curing methods were used; in some methods specimens were wrapped in wax paper and aluminum foil and sealed with tape to prevent evaporation of moisture from the specimens and also prevent contact with carbon dioxide in the air. The oven-cured specimens also were sealed in tin cans. The curing methods are as follows:

1. Wrapped and oven cured at 140 F.
2. Wrapped and oven cured at 110 F.
3. Wrapped and cured at 70± 3 F in a moist cabinet, relative humidity 90 per cent.
4. Cured at 70 ± 3 F in a moist cabinet, relative humidity 90 per cent. Specimens not wrapped.
5. Cured at 70 ± 3 F in a cabinet with relative humidity 45 per cent. Specimens not wrapped.

Effect of Time:

As shown in Figs. 5 and 6, the strength of the lime-stabilized loess increases rapidly at first, then increases more slowly at a more or less constant rate.

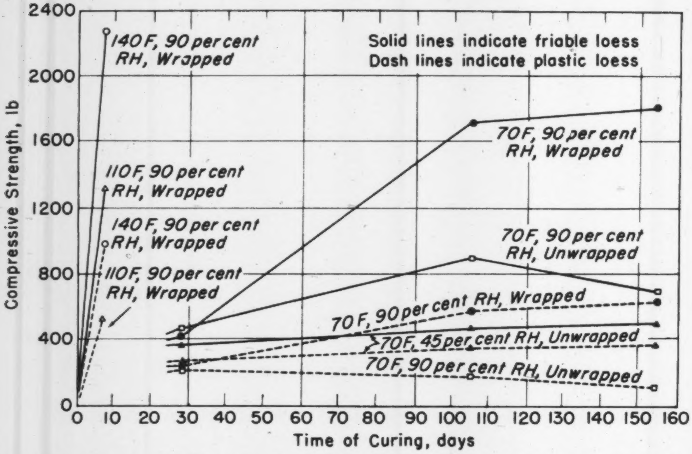

FIG. 5.—Effect of Curing on the Compressive Strength of Loess Stabilized with Hydrated Lime H-1.

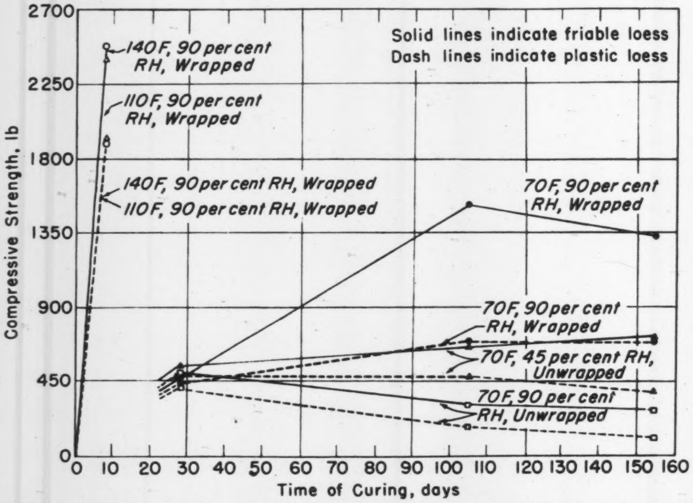

FIG. 6.—Effect of Curing on Compressive Strength of Loess Stabilized with Quicklime Q-1.

After about fifteen weeks the increase is very slight or negative.

Effect of Temperature:

Temperature of curing exerts a tremendous influence on strengths. Seven-day strengths after curing at 110 F or 140 F show a three- to eightfold increase in compressive strength over specimens cured at room temperature. With the plastic loess, 7-day strengths after curing at 110 F are reached in approximately 80 days curing at 70 F. With the friable loess, the 110 F strengths are never reached by normal curing. In no case could 140 F strengths be duplicated by prolonged curing at room temperature. Therefore, accelerated curing, while perhaps useful to indicate relative reactivity, is not recommended for design purposes.

An interesting possibility is that decreased solubility of lime at higher temperatures may account for at least part of the strength increase. Heating a saturated lime solution from 70 F to 110 F will cause a precipitation of 17 per cent of the dissolved lime, and heating to 140 F will cause precipitation of an additional 12 per cent. However, the solubility at 70 F is only 0.164 g per 100 g of water, and heating an ordinary 200-g 2 in. high by 2 in. diam lime-soil specimen to 140 F will cause precipitation of only about 0.05 g of lime. It seems doubtful if the mechanical strengthening from this precipitation would cause an eightfold increase in strength, especially since lime crystals have a weak platy structure. Perhaps the precipitation would give better lime-soil grain contacts and enhance chemical reactions. If this is true, the same effect should be found from alternate wetting and drying of stabilized specimens. The other possibility is that the heat effect is merely a matter of chemical reaction kinetics.

TABLE VI.—CALIFORNIA BEARING RATIO VALUES OF LIME-STABILIZED LOESS.

	Results with Friable Loess				Results with Plastic Loess			
Curing Period	0 day	0 day	7 days	28 days	0 day	0 day	7 days	28 days
Condition at Test	As Molded	Soaked 4 Days			As Molded	Soaked 4 Days		
Admixtures:								
No admixture	12.7	9	9	...	10.5	5	...	...
Calcitic Lime:								
6 per cent H-1	31.5	55	...	...	22.8	31	...	...
4.5 per cent Q-1	34.0	39	...	...	24.0	39	...	...
3 per cent Q-4	...	...	85	88	...	...	...	...
Dolomitic Lime:								
9 per cent H-3	...	...	104	132	...	...	...	66
9 per cent Q-3	...	...	205	215	...	...	84	115

Effect of Humidity:

In all cases specimens wrapped and cured at 90 per cent relative humidity show the greatest ultimate strength gain. It should be noted that this curing procedure would most nearly represent field-curing conditions under an asphalt seal coat. The strength difference sometimes is not apparent until after 28 days, indicating that in damp weather there would be no hurry about applying the seal and there would be little point in temporarily moist curing with wet burlap or straw.

Compressive strengths of unwrapped specimens were usually lower and less predictable. The extreme sensitivity to curing temperature may account for some of the irregularities. In some instances 90 per cent humidity gave higher strengths, in some cases 45 per cent gave higher. Formation of carbonate on the surface of the specimen may aid in sealing the pores against moisture loss. Calcium carbonate crusts were observed to form in the surface layers of specimens after aging about a month. Crusts were observed in specimens molded with sand after curing only three days.

California Bearing Ratio

California Bearing Ratio (CBR) tests were performed with standard Proctor compacted density and according to specifications of the U. S. Corps of Engineers (2). Tests were run immediately after molding, after soaking 4 days, and after moist curing (90 per cent humidity, 70 F) for 7 to 28 days. Results are reported in Table VI. Data are yet incomplete, so comparisons of different limes will not be made. The CBR values now available correlate well with 7-day compressive strength data, and it is believed the same trends will be realized. As an approximation, the CBR value of the friable loess after soaking is about one-fourth the unconfined compressive strength of the 2 in. high by 2-in. diam cylinders; the CBR of the plastic loess after soaking is about one-eighth the compressive strength in pounds.

The values in Table VI indicate a considerable gain in CBR immediately after molding, probably due to cation exchange. The as-molded values are increased two to three times over those of the compacted raw loess. Soaking 4 days further increased the CBR of the lime-treated specimens while decreasing the CBR of the raw loess. In this case soaking of the lime-treated specimens for 4 days is believed to be an effective cure. An additional increase in CBR is evident after moist curing for 7 to 28 days. These strength gains perhaps illustrate the large benefits to be derived from the pozzolanic action discussed later. There was very little or no swelling in the CBR specimens during soaking.

The CBR values now available indicate that loess stabilized with lime would have suitable bearing capacity for use as a base course. Additional criteria are resistance to wetting and drying and resistance to freezing and thawing. Two-by-two in. cylinders were found to gain strength through wetting and drying up to four cycles, probably since the added moisture benefits the cure. Submission of 28-day lime-stabilized specimens to the standard wet-dry test for soil-cement resulted in failure usually by volume change (swelling). Friable loess stabilized with 4.5 per cent quicklime Q-1 showed no adverse volume change and withstood five cycles before the weight loss by brushing exceeded 10 per cent. The severity of brushing will be discussed under freezing-and-thawing resistance; the resistance of lime-stabilized loess to volume change is believed to be greatly influenced by confinement. Lime-stabilized loess, confined as it would be in a road, apparently has little tendency to swell, as indicated by measurements during the 4-day immersion of the CBR test, in which a 10-lb surcharge is used.

According to the Portland Cement Assn. criteria (8) adopted for soil-cement, the freezing-and-thawing resistance of lime-stabilized loess is very low. Specimens usually fail by weight loss during the first two or three cycles. Loss was not by flaking or spalling, as is often true with soil-cement, but by brushing of material from the outside of the specimens. Since cementation of the lime-stabilized specimens is relatively low, it is believed that brushing may be too severe a test. Work with lime - fly ash stabilized soils lends support to this; lime - fly ash - soil mixes which do not pass the brushing test have given satisfactory field performance.[4]

LIME MODIFICATION OF LOESS

The effects of equivalent amounts of limes H-1 and Q-1 on the pH, Atterberg limits, and standard Proctor density were measured immediately after mixing and after 7 days moist curing. Results are shown in Table VII. The plasticity index of the friable loess is only slightly reduced by the addition of lime; the plasticity index of the plastic loess shows a comparatively large reduction. The quicklime was more effective at reducing the plasticity index of both loess samples, although in the case of the friable loess the difference is actually of little practical significance. The shrinkage limit was increased by the addition of lime, the plastic soil showing the greatest increase. Again quicklime was somewhat more effective. On mixing lime with the loess, pH values took an immediate jump to about 12.6 but were reduced during curing, suggesting lime depletion through reactions between loess and lime.

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[4] Oral Statement by Mr. L. John Minnick, G. & W. H. Corson, Inc., Plymouth Meeting, Pa.

Hydrated lime by increasing aggregation decreases the maximum compacted density. The flocculation effect decreases the optimum moisture content about 1 per cent. Quicklime causes about the same reduction in maximum density but raises the optimum moisture content back to its previous level since water is immediately used in the lime hydration. In the mixes given, 1.5 per cent moisture would be sufficient to convert the quicklime.

TABLE VII.—MODIFICATION OF LOESS PROPERTIES BY ADDITION OF HYDRATED LIME OR THE EQUIVALENT AMOUNT OF QUICKLIME.

	Results with Friable Loess					Results with Plastic Loess				
Curing Time	0 day			7 days		0 day			7 days	
Lime	0	6 per cent H-1	4.5 per cent Q-1	6 per cent H-1	4.5 per cent Q-1	0	6 Per cent H-1	4.5 per cent Q-1	6 per cent H-1	4.5 per cent Q-1
LL, per cent	33	34	33	35	33	53	46	41	47	40
PL, per cent	21	24	24	25	25	26	30	30	29	29
PI, per cent	12	10	9	10	8	27	16	11	18	11
SL, per cent	28	29	31	31	31	20	28	32	34	33
pH	7.8	12.6	12.6	12.1	12.0	6.1	12.5	12.7	11.8	12.0
Maximum Dry Density, lb per cu ft	109.9	99.5	97.4	...	...	102.8	94.6	95.1	...	...
Optimum Moisture Content, per cent	18.2	17.1	18.1	...	...	21.4	20.7	21.7	...	...

Reaction of Lime with Loess

It is generally recognized that cation exchange is a mechanism whereby lime can improve the properties of certain soils. However, in the loess samples investigated the natural cations are calcium, and the flocculation mechanism does not appear to be so much an ion exchange as a crowding of additional ions on the clay. This would lower the zeta potential[5] and produce a flocculation. An immediate flocculation effect is observed during mixing and is reflected in lowering of the liquid limit, raising of the plastic and shrinkage limits, and lowering of the maximum compacted density.

In addition to the immediate improvement in properties brought about by flocculation there is a long-term strength gain in lime-stabilized specimens. There has been much speculation as to the reason for this (1, 3, 7) and it appears certain that there is an additional reaction besides flocculation or cation exchange. The high sensitivity of strength gain to the curing temperature suggests a chemical reaction. Goldberg and Klein (3) point out the importance of carbonation of the lime; however, in the present study, specimens wrapped and sealed against the entry of CO_2 gave strengths as high or higher than specimens left unwrapped. Unwrapped specimens definitely showed the development of a carbonate-rich layer around the outside.

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[5] The zeta potential is a measure of the effective negative charge at the surface of the clay. The addition of positive ions will reduce the charge.

For long-time strength gain, a pozzolanic reaction has been suggested, and it is usually supposed that this reaction takes place with the clay. However, in this investigation higher strengths were realized with less clay, and there was little difference in optimum lime content between high clay and low clay. The same trend was reported by Goldberg and Klein; a montmorillonite containing 40 to 50 per cent nonclay minerals gave stabilometer values several times those obtained with a nearly pure montmorillonitic clay. These percentages of clay are so high as to minimize the influences of granulometry; a soil containing 50 per cent clay could allow little contact between the silt grains. This would suggest that important reactions may occur between lime and the non-clay minerals. Petrographic and soil-cement studies with loess show that strength correlates with quartz surface area (**4**, **5**). It, therefore, appears that pozzolanic reactions may be most pronounced with the nonclay minerals and particularly with quartz, so long as the particle size is fine enough. A more detailed discussion of the reactions and a possible explanation for the influence of the calcium: magnesium ratio will be found in reference (**4**).

CONCLUSIONS

The laboratory data indicate that a field trial of lime-stabilized western Iowa loess would be desirable. However, the laboratory results certainly point out the desirability of evaluating a lime prior to its use for stabilization. With Iowa loess, a dolomitic lime would probably be the more effective. Quicklime, even though requiring more caution in handling, may offer the best and most economical way for lime stabilization. It is not yet known if tank-truck hydration of the quicklime will yield the same results as mixing it dry with the loess. With proper information on the lime and on the soil, the most desirable properties of lime for a certain job may eventually be predicted; it appears that the main deterrent at the present time is lack of information of the lime. A simplified pozzolanic reaction rate test would therefore seem desirable.[6]

Resistance of lime-stabilized loess to freezing and thawing can best be evaluated by means of field trials, after which suitable laboratory test criteria might be developed. Curing is best at high temperatures, so early summer construction seems desirable. Moist conditions should be maintained for proper curing, for example by the use of a bituminous seal applied immediately after finishing the base. Strengths attained in the laboratory by accelerated (high-temperature) curing may never be reached under normal field conditions and so should not be used in design.

Acknowledgment:

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Special thanks are due to Mr. R. S. Boynton, General Manager of the National Lime Assn and to member companies for furnishing lime, and to Mr. L. W. Lu of the Iowa Engineering Experiment Station for contributing some of the CBR data.

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[6] Such a test is now being developed in Iowa State College Laboratory.

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DISCUSSION

Mr. L. John Minnick.[1]—Were any tests carried out to determine the effect of carbonation due to the presence of CO_2 in the atmosphere? We have found in running similar tests to those reported in this paper that carbonation has a bearing on some of the results.

Mr. J. G. Laguros (*author*).—Special tests along that line have not been performed, but in many instances when the specimens were tested it was found that with prolonged curing the specimens developed a crust, so to speak on their surface. This is strong evidence of carbonization. The crust, which at times assumed the form of a flakey surface, appeared within three days in some cases, and in others it did not appear until after 6 months. Experiments with sandy soils indicated that the formation of the crust took place very rapidly.

Mr. Robert S. Boynton[2] (*by letter*).—The authors are to be complimented for a very comprehensive research study on lime stabilization, and they have exhibited some new and ingenious thinking on this subject. In general the results they have obtained on compressive strengths, California Bearing Ratio (CBR) values, and physical constants of soils, in which comparisons are drawn between raw loess soils and varying percentages of lime, are quite similar to the results obtained by Purdue University, University of Texas, and the highway departments of Texas, California, Louisiana, Virginia, and other organizations which show marked changes in clay and certain silty soils by the addition of lime. The only major exception is that Iowa has found dolomitic limes to be uniformly more effective in varying degrees than high-calcium limes—at least on these two particular Iowa loess soils.

The greater equivalent effectiveness of quicklimes over hydrated limes found by Iowa is not surprising and confirms other research findings on this subject. It is believed that this phenomenon is largely due to the heat of hydration created when the quicklime is slaked in the soil after water is added. This accelerates the set and resultant strength of the lime-soil mixture in the same manner as when the samples were oven heated during laboratory curing.

Both high-calcium and dolomitic limes have been successfully used for road stabilization, and very little difference, if any, has been detected in their relative effectiveness. Actually, since the production of high-calcium lime is more widespread and greater in tonnage than dolomitic, much more of the high-calcium type has been actually used in road construction. In Texas and other southwestern states where only high-calcium lime is produced, it has been the only type used. With certain clay soils, usually the gravelly type, some spectacular improvements have been obtained with 3 per cent of lime by weight both in laboratory and field control testing. Raw soils having a CBR value of 6 have been increased to 90; the same and other soils having a plasticity index of 40+ have been reduced to 8. The implication of this paper that dolomitic lime might further improve such results is slightly incredible.

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[1] Vice President in charge of Research, G. & W. H. Corson, Inc., Plymouth Meeting, Pa.

[2] General Manager, National Lime Association, Washington, D. C.

In attempting to rationalize the reason for this Iowa result which is contrary to other thinking, the following thoughts occur. The high-calcium hydrate is completely hydrated, whereas the type N dolomitic hydrate has only the calcium and a small portion of the dolomitic components hydrated. It is 25 to 30 per cent unhydrated. The unhydrated portion is in oxide form as MgO. Since the oxide (quicklime) form has proved to be more effective than the hydroxide (hydrated) form, the dolomitic hydrate presumably contains more of the more active oxide ingredient. Yet, this theory breaks down on Iowa's findings that dolomitic quicklime was more effective than high-calcium quicklime, both of which have an equal oxide content.

It is noted that small specimens of 2 by 2 in. were used. Other investigators have molded larger specimens of from 4 by 4 in. to 6 by 8 in. It is believed that more accurate and consistent results are obtained in testing the larger specimens. At best there is certain to be a normal discrepancy in laboratory results, but possibly this variance in values is accentuated with the smaller specimens. For example, in Fig. 3 it is rather paradoxical and erratic that the 1:3 ratio of calcium and magnesium is intermediate with the calcium and magnesium ratio of 3:1 and 9:1. This plus the fact that there may be something unique in the chemical composition of these Iowa loess soils that lend themselves better to dolomitic limes is an offered explanation.

This writer feels that generally the greatest variable in lime stabilization occurs with the countless different soils and that there is very little difference in the effectiveness of commercial limes of both high-calcium and dolomitic types, and that trying to evaluate their effectiveness in a laboratory would be nearly as inconclusive as with portland cement. This is not true with waste limes which contain much higher contents of carbonates and silicates and which dilute the active component of the lime.

In general, Iowa's results showed somewhat higher early strengths (7 days) and lower ultimate strengths than other investigators. Laboratory specimens and cores from lime stabilized roads have almost invariably shown a slow, steady increase in strength up to 2 to 3 yr.

Iowa's findings that the pH of the laboratory specimens were sharply reduced during curing is new and interesting. It adds further evidence that a pozzolanic reaction occurs in the formation of monocalcium or magnesium silicates since such combined lime compounds possess a pH much lower than uncombined lime.

MR. F. N. HVEEM.[3]—This interesting paper throws additional light on some of the problems and variables involved in improving soils by additions of lime. From one viewpoint it might be considered that the results reported are at variance with those obtained in work here. However, it seems more likely that the differences derive from differences in the soil types involved.

Taking up a few points in the paper, I offer the following comments:

It is stated that for the stabilization of loess "the ideal lime composition appears to be between 1:1 and 3:1, Ca:Mg." In our own experience with bentonitic and kaolinitic soils, pure $Ca(OH)_2$ performed as well and perhaps better than a dolomitic lime meeting the above limits of composition.

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[3] Materials and Research Engineer, California Division of Highways, Sacramento, Calif.

The authors note that "On the basis of compressive strength, quicklime appears to be more effective with loess than the equivalent amount of hydrated lime..." On the basis of R-value tests on our soil materials, we found no appreciable difference between the results obtained with quicklime as compared with hydrated lime.

The authors note that "dolomitic lime is more effective than calcitic lime (with loess)." On the basis of R-value tests, all limes gave similar strengths with the exception that a locally available dolomitic lime gave lower strengths with both bentonite and kaolinite. Our results are therefore not in agreement with those reported by the authors.

The authors state "Among the different variables affecting the strength of lime-stabilized loess, curing is of major importance. Its effect on strength is a function of time, temperature, and relative humidity . . . the strength of the lime-stabilized loess increases rapidly at first, then increases more slowly at a more or less constant rate. After about fifteen weeks the increase is very slight or negative." Although we have not measured the effects of varying temperature and humidity, we have found that curing time does have an effect, especially with $Mg(OH)_2$. Incomplete and scattered data suggest that $Ca(OH)_2$ develops virtually its total strength in a short time; say within seven days; whereas $Mg(OH)_2$ may require several months to reach its maximum value.

In conclusion, we can only say at this time that there appears to be points of agreement and points of divergence between their work and that carried on here. It seems quite possible that all or a part of the differences noted may be due to the inherent differences between loess and clay. However, this does not rule out the possibility that there are also differences in the limes used and some difference in evaluation may derive from the fact that our tests were based on R-value comparisons rather than compressive strength.

MESSRS. J. G. LAGUROS, D. T. DAVIDSON, AND R. L. HANDY (*authors' closure*).—The interest and discussion in this paper are sincerely appreciated and have pointed up some interesting possibilities. Further studies have shown that while lime carbonation is indeed important, as suggested by Minnick, it probably is not a major factor differentiating calcitic from dolomitic lime strengths; the same relative strength differences are found in specimens stored in the open or completely sealed from the atmosphere. However, to avoid variable carbonation all Iowa soil-lime specimens for long-term curing are now double wrapped in waxed paper and aluminum foil.

The authors would like to vindicate themselves and point out that the purpose of this work is not to suggest a low effectiveness of calcitic limes so much as to illustrate the higher strengths possible with certain dolomitic limes. Higher strengths are necessary if soil-lime is to withstand freezing in northern climates, and as yet very little successful usage of lime for base course stabilization has been reported from this area. On the other hand, lime stabilization has a long history of successes in the South.

Our laboratory work has now progressed with other soils with both lime and lime - fly ash stabilization; soils tested include other Iowa soils and soils from other states and Alaska with montmorillonitic, illitic and chloritic clay minerals. Monohydrate dolomitic lime nearly always gives higher strengths, and there appears to be nothing unique about Iowa loess in its reactions with lime. However, strength differences are less with clayey soils. Some of these data on soil-lime have been reported to the Highway Research Board.[4] That study revealed that a more important variable was the brand of lime. Apparently some commercial hydration processes result in partial hydration of MgO, reducing dolomitic lime reactivity to the approximate level of calcitic lime. Where hydration is controlled and heat and pressure are eliminated, as in hydration of dolomitic quicklime mixed 1:20 with soil, strengths closely correspond to those realized with synthetic limes prepared from pure chemicals.

The question of heat of hydration of quicklime contributing to strength may be resolved by use of a thermometer. A small sample size, low lime contents, and use of metal mixing bowls and mechanical mixers in the laboratory for 5-min mixing periods result in heat dissipation prior to molding. The effect is therefore believed to be small.

If height-diameter ratio is ignored, ideal specimen size is related to the coarsest material present. A small specimen size has been selected for these tests partially for convenience and conservation of materials but mainly because of the superior reproducibility of strength results. The wide use of 2-in. diameter specimens in this country and abroad for fine-grained soils is perhaps an endorsement of this fact. An advantage of the 2 by 2-in. size is that uniform density can be obtained from compaction in a single layer, thus avoiding stratification within a specimen. With coarse materials, of course, use of a larger specimen size is unavoidable.

Data for intermediate Ca:Mg ratios as in Fig. 3 show that while the curves indeed seem paradoxical, the complete data fit on a smooth curve which peaks at a Ca:Mg ratio of 1.0. The completed ratio *versus* strength curve will be included as Fig. 3 of the later paper.[4] For a more complete discussion of reasons one should refer to that paper.

So far as the effect of curing time on strengths with the various types of limes, the authors must plead even more incomplete and scattered data than does Mr. Hveem. The excellent remarks Hveem makes on this subject are certainly worthy of careful attention and consideration. We have no data for direct comparison of dolomitic dihydrate and calcitic limes, since our work is now mainly with monohydrate lime. However, our data do show that the long-term strength gain with MgO lime is greater than with $Mg(OH)_2$. For example, the increase in flexural strength from 7 to 84 days is 350 per cent for friable loess with 9 per cent dolomitic quicklime; with dihydrate dolomitic lime the 7-day strength is lower, and the strength increase from 7 to 84 days is only 230 per cent. Probably the MgO acts as MgO—it does not hydrate before becoming active with $Ca(OH)_2$, water and soil.

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FIELD AND LABORATORY CORRELATION OF THE BEARING CAPACITY OF HARDPAN FOR DESIGN OF DEEP FOUNDATIONS*

By W. S. Housel[1]

Synopsis

This paper presents the essential steps in the continued development of the penetration method of evaluating soil resistance presented to the Society in 1935 (**1**)[2] supplemented by a parallel development in field loading tests and laboratory shearing resistance tests, including ring shear, unconfined compression, and triaxial compression. The latter test followed concepts of internal stability of granular material presented to the Society in 1936 (**8**).

Results are reported under the following subdivisions:

1. The Michigan penetration index as a field measure of soil resistance.
2. Correlation of the Michigan penetration index with the Raymond penetration, and conversion factors involved in the use of either method.
3. Laboratory shearing resistance tests and their correlation with field penetration and field loading tests.
4. Methods of evaluating soil resistance factors involved in granular-cohesive mixtures, and estimating the bearing capacity of hardpan.
5. Test procedures and methods of analysis for field loading tests on piles.
6. Comparison of computed and measured values of the bearing capacity of hardpan for design of deep foundations.

Data from large scale field loading tests provide the primary basis for establishing the validity of the tests under practical conditions. As a result of this work, the applicability of the penetration method as a rapid determination of soil resistance has been extended and amplified until today it is being used with increasing confidence.

Beginning in 1927, a program of soil investigation involving both field and laboratory testing was undertaken by the University of Michigan in cooperation with a number of other organizations. These participants have included many other public agencies as well as engineering organizations and engineers in private practice. Soil investigation and research made possible by this program have been a part of the engineering design studies on more than 600 construction projects of varying magnitude, over a period of more than 25 yr.

This paper reviews and summarizes the results of some of this experience, with particular attention to testing procedures developed for both field and

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] Professor of Civil Engineering, University of Michigan; Research Consultant, Michigan State Highway Dept.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1346.

laboratory determination of soil resistance. The studies cover a wide range of soil conditions and design requirements. Data are presented on the correlation of field penetration measurements, laboratory shear tests, and full scale field loading tests on both friction and bearing piles. The penetration measurements include those developed by the author and reported to the Society in 1935 (1), and the penetration test most widely used in commercial boring practice, developed by the Raymond Concrete Pile Co. The laboratory shear tests include ring shear, unconfined compression, and triaxial compression tests. While some data on friction piles are included, the emphasis is on field loading tests on high-capacity bearing piles which involve the bearing capacity of hardpan.

The material identified as hardpan in the Detroit area requires definition, as the local terminology may not be generally recognized. Hardpan in Detroit is the remnant of the Illinoian drift immediately above bedrock, left in place and consolidated by the last or Wisconsin ice sheet. It generally consists of a highly indurated mixture of sand, gravel, and clay, and may be more accurately termed claypan. From the standpoint of soil resistance, it is a granular-cohesive mixture with high cohesion and a dense granular structure with high internal stability. In some cases, this deposit contains bodies of sand, gravel, and boulders, with very little clay binder. However, these granular materials are generally highly consolidated, and when confined by 100 ft or more of overburden, they provide supporting capacity that may be regarded as unusually high.

PENETRATION MEASUREMENTS

The development and refinement of penetration observations have gone through several stages, and it is only in the last ten years that their full potentialities have been realized. Now, after repeated demonstrations supported by full scale field loading tests, engineers are gaining confidence in their use. As previously noted, there are two types of penetration measurements that have been used in these studies. The developments involve the correlation of these two methods and the determination of conversion factors so that either one may be translated into terms of soil shearing resistance and internal stability.

University of Michigan Penetration Method:

The penetration method developed as part of the University of Michigan research program has been described in detail (1), and only brief references to the previous publication will be made to serve as a background for the further development. Observations are made of the number of blows required to drive a standard core barrel a depth of 6 in., and this has been defined as the penetration index, N. The driving is done with a falling weight of 20 lb, with a height of fall of 34 in. The dead weight, which consists of the core barrel, driving head, and connections, is standardized at 20 lb.

The major difference between this penetration test and those generally used in commercial boring operations is the control of the dead weight as a constant. In commercial boring practice, the dead weight varies through a wide range, including as it does a variable length of drill rod or pipe. In order to maintain a constant dead weight, the entire driving mechanism must be lowered to the elevation at which the sample is being taken. In Fig. 1 are shown three types of penetration devices that have been used in the University of Michigan investigations. Figure 1 (*a*) is the simplest driving mechanism of all, for use in test pits or on highway subgrades where the sampling elevation is accessible. Figure 1 (*b*) shows

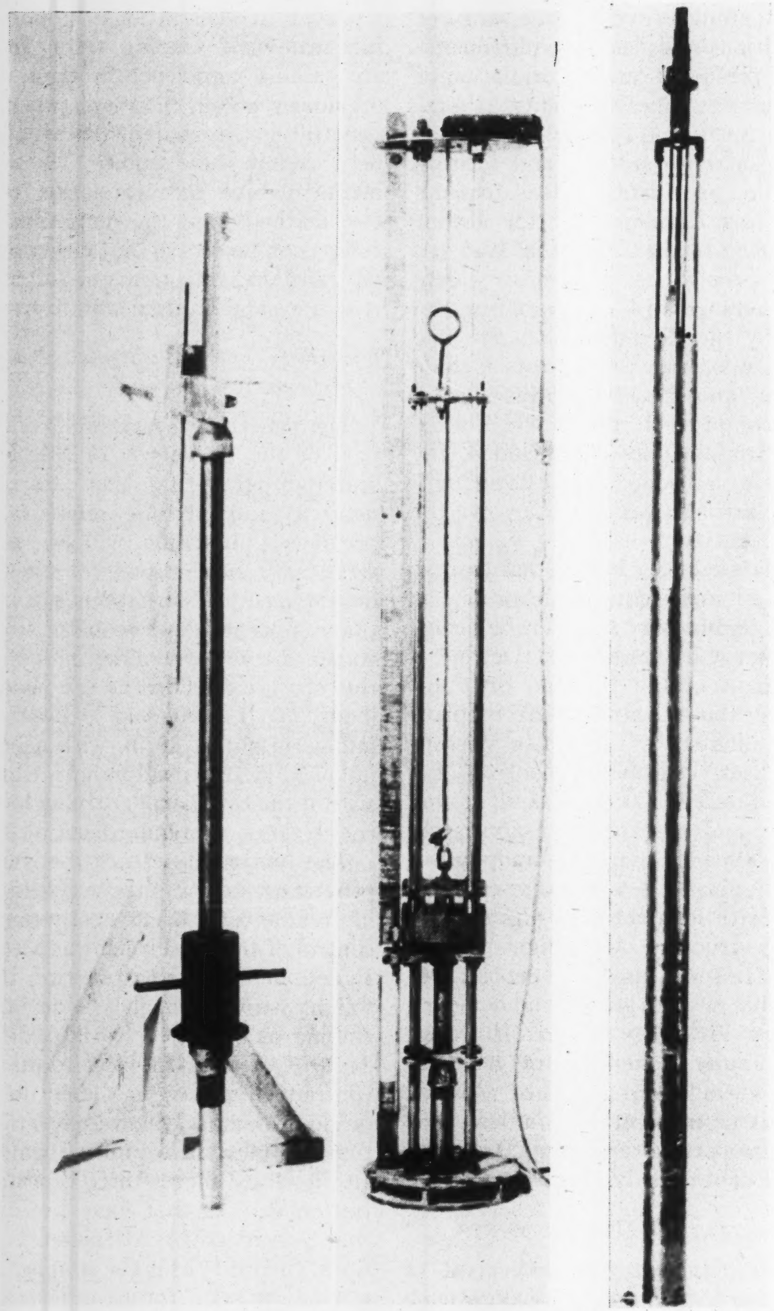

FIG. 1.—Types of Michigan Penetration Devices.

one of the earlier models of the penetration apparatus which could be lowered into a 12-in. casing for sampling at any depth. In Fig. 1 (*c*), the driving mechanism is encased in a 4-in. cartridge for sampling at greater depths. This model was used in the foundation investigation for the Mackinac Straits Bridge, where undisturbed samples of clay overburden were taken in depths of water and soil up to 170 ft.

Evaluation of resistance to penetration of an open core barrel, and correlation of this resistance with the static shearing resistance as measured by laboratory tests were presented in considerable detail in the above reference (1). This and other publications also gave correlations between the penetration index, N, and bearing capacity determined from plate loading tests (2). Additional data are now available, and the primary objective at this time is to present additional developments, reviewing prior results only briefly to serve as a background.

Equation 1 is the basic relation developed for evaluating the force, F, resisting penetration under the assumption of plastic impact:

$$F = \frac{hW^2}{2p(W + w)} \qquad (1)$$

where:

F = force in lb,
W = falling weight in lb (= 20 lb),
w = dead weight in lb (= 20 lb)
h = height of fall in in. (= 34 in.), and
p = penetration per blow in in.

Through extensive observations and statistical analyses, the force resisting penetration was translated into terms of point resistance and side wall shear or friction leading to the following equation, in which S_c is shearing resistance in pounds per square inch, and N is the penetration index, as defined in the Michigan method:

$$S_c = 0.593\, N^{1.01} \qquad (2)$$

In the later development of the penetration method there were some minor changes in the dimensions of the core barrel, which caused some slight adjustment in the numerical factors in Eq 1. Continued correlation of the equivalent shearing resistance from penetration with laboratory shear tests and other independent tests provided a more reliable determination of the relation between the static shearing resistance of the soil and the resistance to penetration under dynamic impact. An impact factor, C_I, was introduced to represent variation in dynamic reactions involved in the penetration observations (3). Equation 2 was simplified to the following form:

$$S_c = 1.2\, C_I N \qquad (3)$$

The impact factor, C_I, varies with the dynamic characteristics of the driving apparatus and should be determined by calibration for each individual machine or type of machine. Assuming a free fall and perfectly elastic impact, C_I approaches unity as the limiting value, while the value of C_I for plastic impact is $\frac{1}{2}$, a value which corresponds with the original development of Eq 1. Continued experience with correlating penetration observations has led to the conclusion that when sampling at considerable depth, the impact approaches plastic impact and a value of C_I of $\frac{1}{2}$. Correlation with the subgrade penetrometer indicates a value of C_I of approximately $\frac{3}{4}$, which represents present practice. With these values of the impact factor, the static shearing resistance of cohesive clay is estimated from the penetration index, N, by the following equations, with the shearing resistance, S_c, in pounds per square inch.

Subsurface borings, $S_c = 0.6\,N$ . . . . (3*a*)

Subgrade penetrometer, $S_c = 0.9\,N$ . . (3*b*)

Expressed in the customary unit of pounds per square foot (psf), the range for the shearing resistance, S_c, estimated from penetration measurements is as follows:

$$S_c = 86\,N \quad \text{. . . . . (3a)}$$

$$S_c = 130\,N \quad \text{. . . . . (3b)}$$

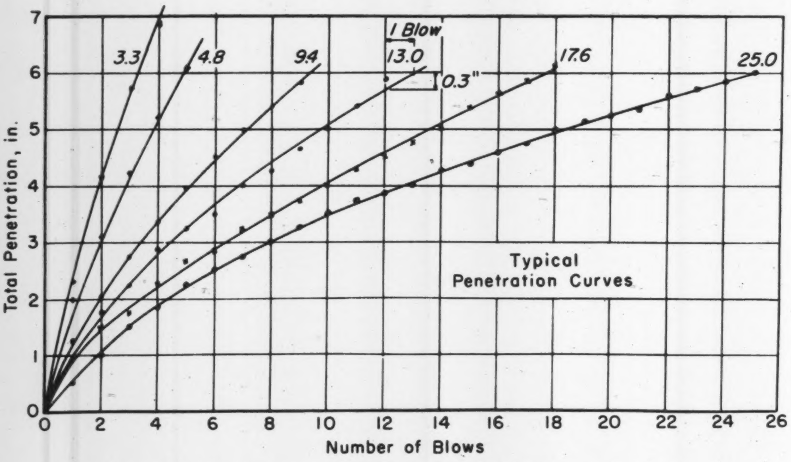

FIG. 2.—Typical Penetration Curves by the Michigan Method.

By far the greater volume of penetration observations have been in connection with samples from borings. In later years, most of this sampling has been done by contract with the Raymond Concrete Pile Co. using their standard penetration measurements. The Raymond penetration observations involve several substantial variations from standards employed to establish the University of Michigan penetration index, *N*, as outlined above. In the first place, the sampler is driven with a 140-lb weight falling 30 in., which is a much heavier blow. On the other hand, the standard length of drive is 12 rather than 6 in., and the dead weight of sampler and drill rod is much greater and varies with the depth of the boring. Empirical relations have been worked out for reducing Raymond penetrations to the University of Michigan penetration index, as outlined below. These estimates of shearing resistance and, in fact, all penetration measurements taken in borings by presently used equipment are now translated into shearing resistance in pounds per square foot in terms of Eq (3*a*).

Standardizing Variable Penetration Observations:

In order to use penetration measurements as they are ordinarily taken in current practice, it is necessary to provide a means for reducing such measurements to an established standard. There are several variable factors involved; (*a*) the depth of penetration or length which the core barrel is driven may vary for many reasons; (*b*) the driving force of the blow, usually expressed in terms of a weight and height of fall, may vary; and (*c*) the dead weight of the sampling device varies through wide limits when the sampling is done at variable depth with corresponding lengths of drill rod or pipe.

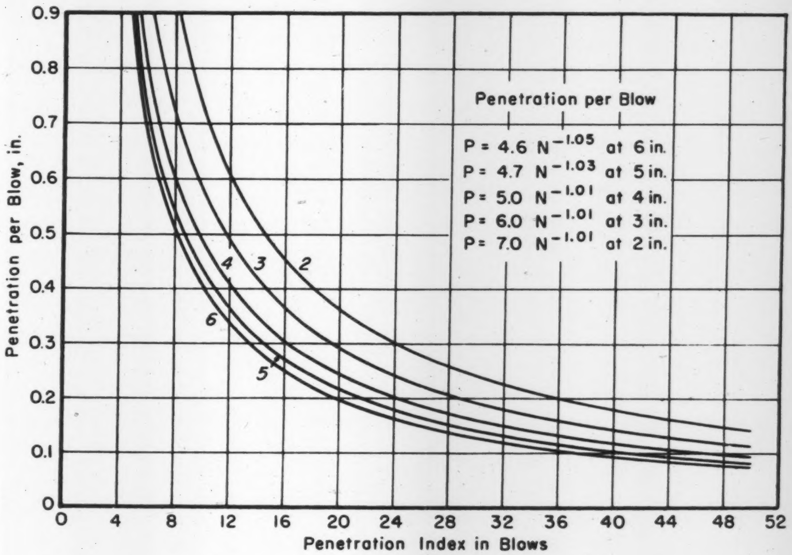

FIG. 3.—Penetration per Blow *versus* Penetration Index.

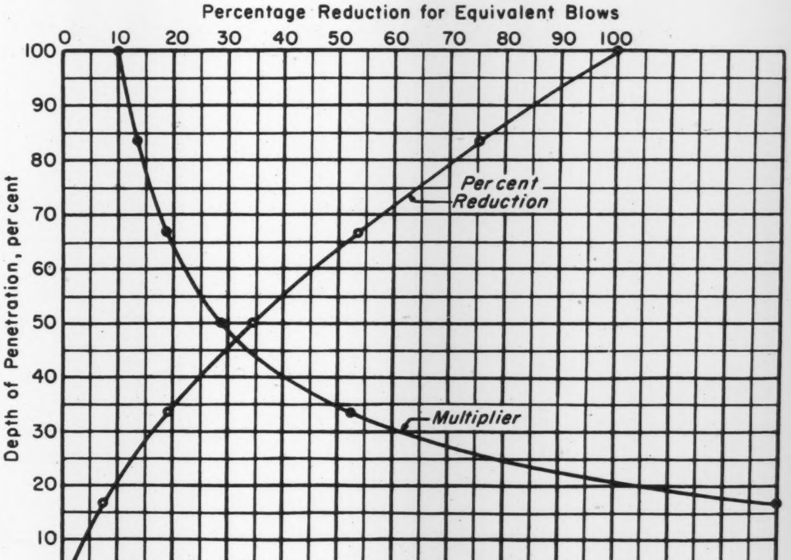

FIG. 4.—Conversion Factors for Equivalent Penetration.

In the original development of the penetration method, it was shown that for any given soil there was a definite relation between the number of blows for each inch of penetration and the penetration index, *N*. From these relations, which are illustrated in Figs. 2 and 3, it is possible to determine the number of blows required to convert from any one depth of penetration to another. These data have been plotted in Fig. 4, which shows the depth of penetration in terms of a percentage of any total penetration and a corresponding percentage of the number of blows for any fractional penetration. A second curve is shown giving an inverse relation as a multiplier which can be used to determine the number of blows required to increase the depth of penetration to a selected standard.

The two following rules may be used as instructions for the use of the conversion factors represented in Fig. 4.

Rule 1: To obtain the number of blows required to increase a smaller to a greater penetration, multiply the number of blows recorded for the fractional penetration by the multiplier from the curve at the given percentage of penetration.

Example: If 8 blows were required for a penetration of 5 in., what is the penetration index, *N*, for the established standard of 6 in. penetration?

Penetration percentage = $\frac{5}{6} \times 100 =$ 83.8 per cent.

Multiplier = 1.4.

Penetration index, $N = 1.4 \times 8 = 11.2$ blows.

Rule 2: To obtain the number of blows for a fractional part of a total penetration greater than required, reduce the total number of blows by a percentage reduction on the curve at the required percentage of the total penetration.

Example: When the number of blows reported for a penetration of 12 in. is 40, what would be the reduced number of blows for 6 in. penetration?

Penetration percentage = $\frac{6}{12} \times 100 =$ 50 per cent.

Percentage reduction in blows = 34.5 per cent.

Reduced number of blows = $0.345 \times 40 = 13.8$.

The conversion of Raymond penetrations to the University of Michigan penetration index also involves the magnitude of the blow and the inertia of the dead weight of drill pipe and sampler. In practice, these two variables are combined in a single empirical factor which modifies the impact factor, C_I, used in the original development of the University of Michigan Standard.

As previously noted, the blow of the weight of 140 lb falling 30 in. is obviously much heavier than that of the 20 lb weight falling 34 in. used in the Michigan penetrometer. In many cases, however, this may be more than offset by the inertia of the dead weight and other impact-absorbing factors involved in sampling at considerable depths below the surface. Studies made by the Bureau of Reclamation laboratory in Denver (**4**), which were somewhat preliminary in nature, indicate that in cohesionless soils, overburden pressure and relative density are the most important factors, but increased length and weight of drill rod and increased weight of the sampling device do have a measureable effect in increasing the apparent resistance to penetration.

In the Michigan studies, the conversion of Raymond penetration to equivalent shearing resistance in cohesive materials, or internal stability in granular materials, has been most definitely established in determination of the bearing capacity of hardpan in the Detroit area. There have been a number of major building projects involving the design of caissons or bearing piles, for which such studies have been made. In these cases, the Raymond penetrations are from preliminary borings in which the samples have been taken at depths of approximately 100 to 120 ft. Later, during construction, the penetration resistance has, in some cases, been checked in the bottom of the caissons with the Michigan penetrometer. In other cases, full scale loading tests on end-bearing piles have been made to provide an independent measure of bearing capacity and to check the estimates made from penetration measurements.

The opportunities for a direct comparison between the Michigan and Raymond penetrations consist of two projects in Detroit, where the Michigan penetration index was measured and standard samples were taken during construction at the bottom of caissons. The first such project was in 1947–48 for an addition to the Conners Creek power plant of the Detroit Edison Co. The second was in 1951 for the City-County Building, a major office structure consisting of a 20-story office unit and a 14-story courts unit. Caissons were used in both structures, bearing on hardpan at a depth of approximately 120 ft below ground surface.

At the Conners Creek plant, the initial borings were made by the Raymond Concrete Pile Co., using their standard driving procedure. Subsequently, three sets of Michigan standard samples were taken in two test caissons at two elevations in hardpan. The hardpan was then checked in 78 caissons during construction, with the penetration index being used to control the depth and bell diameter of the caissons. At the City-County Building, there were two sets of preliminary borings, one by the City of Detroit Soil Testing laboratory and one by the Raymond Concrete Pile Co. Subsequently, the Michigan penetrometer was used to measure the penetration index and obtain samples for laboratory shear tests in 126 caissons. The penetration index was again used to check the bearing capacity of hardpan in each

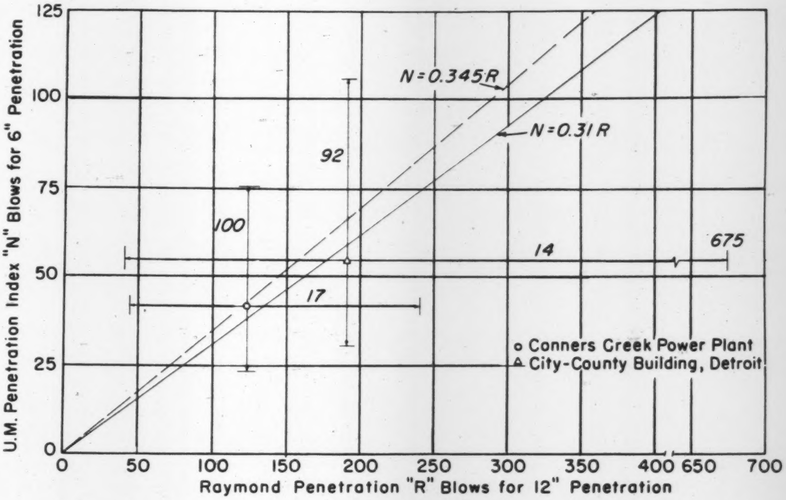

FIG. 5.—Field Observations of Michigan and Raymond Penetrations.

caisson and to control the depth and bell diameter, both of which were varied to provide uniform support throughout the building.

In Fig. 5, in which the Michigan penetration index is plotted as the ordinate and the Raymond penetration as the abscissa, the full range of variation in both types of penetration is shown by the length of the indicating arrows, and the average of the respective ranges is indicated by the plotted points. The number shown in connection with each observation is the number of samples involved in each determination. There are two straight lines shown for comparison, which represent the factors used in converting Raymond penetrations to the Michigan penetration index. The dashed line represents a percentage reduction in blows from the 12 in. drive used by Raymond to the 6 in. drive used by the Michigan laboratory.

At the depth of 100 to 120 ft to hardpan, the heavier blow in the Raymond sampling is almost completely compensated by the inertia and other impact absorbing characteristics of more than 100 ft of drill pipe. Based on the average penetration index as measured by the Michigan penetrometer at the bottom of the caissons, an added correction factor of 0.9 was applied to convert Raymond penetrations to the penetration index, N. This final conversion factor is shown as the full straight line in Fig. 5, which may be expressed by the following equation in which R represents the Raymond penetration:

$$N = 0.9 \times 0.345\ R = 0.31\ R$$

It should be emphasized that this conversion has first been established for sampling in borings and caissons at a depth of 100 to 120 ft. It depends upon a relatively small number of observations from the borings on the two projects cited, which provide the only direct comparison between the two penetration methods available at this time. There are a number of other sources of experimental data corroborating the above relationship between the two types of penetration measurement and establishing their relationship to other methods of measuring soil resistance. The presentation of these data in considerable detail is the purpose of this paper, and they will include the following methods to be compared and correlated.

1. Laboratory shear tests
    - *a.* Ring shear
    - *b.* Unconfined compression
    - *c.* Triaxial compression
2. Field loading tests
    - *a.* Soil bearing or plate tests
    - *b.* Friction or shear piles
    - *c.* Point bearing piles to hardpan

1. LABORATORY SHEAR TESTS

The essential features of the test procedures used are given below:

Ring Shear Test:

The shear test that has been most closely identified with the University of Michigan Soil Mechanics laboratory has been called the ring shear test. It has been in constant use at Michigan since 1934. In the past 22 years, this laboratory has conducted some 25,000 such tests on all projects.[3] The test procedure has been published in detail (**3**, **5**, p. 286), and its practical application has been described in other publications (**6**, **7**). So far as is known the ring shear test has been adopted in one form or another by 11 soil mechanics laboratories.

One of the essential features of the test is its adaptability to commercial boring practice with a $1\frac{3}{4}$ to 2-in. core barrel and inner steel liners easily removable for shipping and laboratory testing. This permits the testing of the soil core in double transverse shear without removal or handling of the soil core itself. The second and most important feature of the Michigan test is the method of increment loading at constant time intervals. This permits determination of the relationship between loading and rate of settlement necessary to measure the true yield value of the soil.

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[3] Includes 347 projects by the Michigan State Highway Dept.

Thus, the ring shear test is a measure of what may be called the static yield value or shear stress greater than which the soil will suffer progressive deformation. In these tests, observations are made of the rate of shearing deformation for each load increment applied. From this may be determined, by extrapolation, the actual load at which progressive deformation begins. The final results as shown are thus independent of dynamic resistance and represent that applied stress which may be sustained in static equilibrium.

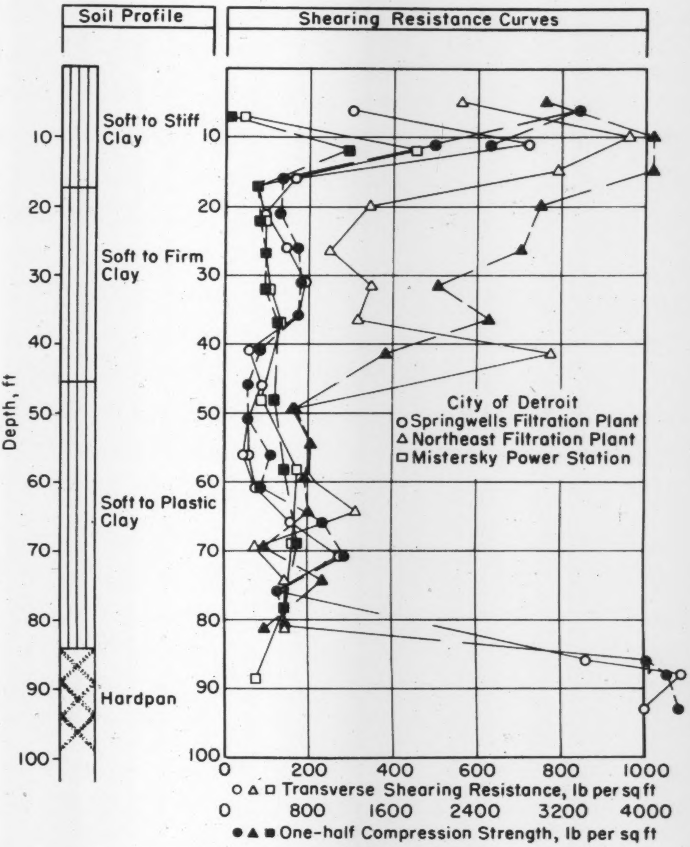

FIG. 6.—Typical Shear Profiles.

Unconfined Compression Test:

Starting in 1942, the Michigan laboratory has conducted an unconfined compression test in parallel with the ring shear test. This practice was adopted because of frequent collaboration with other laboratories where the wide disparity in shear test results caused difficulty and misunderstanding. Under generally accepted procedure, the load in the unconfined compression test is applied at a continuous rate until failure is produced and in a much shorter period of time (5 min loading period). Over several thousands of parallel tests, it has been found that shear values obtained from these unconfined compression tests are very close to four times those obtained from the ring shear tests. This is particularly true in the case of plastic clays of the truly cohesive type or in the case of saturated clays. It has also been found that for stiff clays and for materials which have granular characteristics, the ratio of the shear values obtained from these two tests is somewhat erratic, and, in general, higher than in the case of plastic clays. In presenting the results from the unconfined compression, it has been the practice to plot these shear values to a scale four times greater than is used for the ring shear test, which in most cases brings the divergent results into close focus.

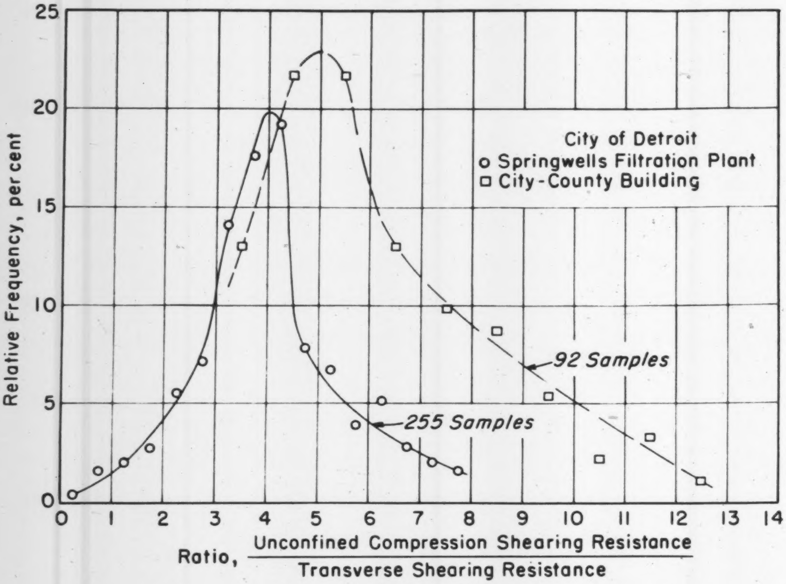

FIG. 7.—Statistical Analysis of Shear Test Results.

The unconfined compression test has been described in detail (3, 5, p. 253), and only results are now to be given. Since 1942, the Michigan laboratory has conducted some 22,800 such tests on all projects. These tests have been on the same samples for which the field penetration was observed and ring shear test value determined. A direct comparison between the transverse shear from the ring shear test and the compression shear from the unconfined compression test is given in Fig. 6, which shows typical shear profiles from three different projects. Attention is again directed to the dual scale and the fact that the equivalent static shear value from the compression test is only $\frac{1}{4}$ of the value observed under the rapid rate of loading.

The data in Fig. 6 have been selected to illustrate the two important observations given in the preceding paragraphs that can now be supported by results from thousands of tests. In all three projects, the agreement with the four to one ratio is very close in the soft or plastic clays, and it is substantially larger and more erratic in the stiff clays affected by granular structure.

The statistical analysis of data such as these has been extended to further evaluate the ratio between compression shear and direct shear. In Fig. 7 are shown two probability curves in which the ratios of compression shear to direct shear have been plotted as the abscissa, and the percentage of the total number of samples tested is plotted as the vertical ordinate. These are job curves from which the most probable value of the shear ratio is determined. In the case of the Springwells plant, a shear ratio very close to 4 is indicated; while in the case of the City-County Building, the most probable value is 5.0. It may be noted that the number of samples involved in these two jobs is sufficient to establish a fairly adequate basis for statistical analysis, there being 255 and 92 samples, respectively. This is a condition which must be met to eliminate the effect of the wide range of variation commonly found in individual soil tests and to provide a representative measure of soil resistance in the entire mass.

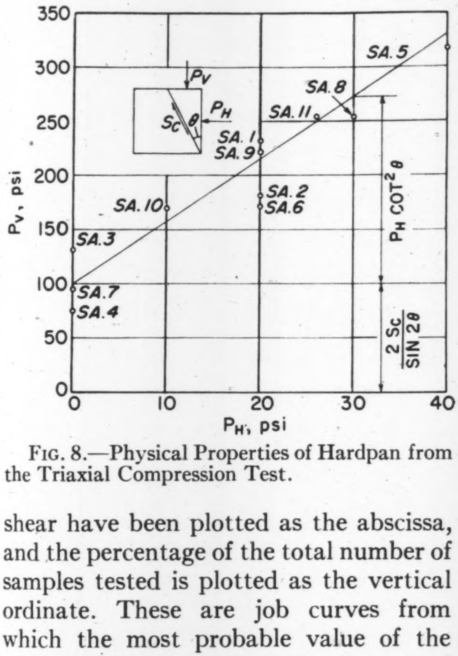

FIG. 8.—Physical Properties of Hardpan from the Triaxial Compression Test.

Triaxial Compression Tests:

The determination of the bearing capacity of hardpan requires more than a measure of shearing resistance, as it is a granular-cohesive mixture in which internal stability of the granular structure is a controlling factor. In 1936 (**8**), the author presented a new concept of internal stability based on several years of experimental research on granular materials by Berry (**9**), which included what was then called a stabilometer test (**2,5**, p. 315).

The mechanical advantage provided by a granular structure was clearly differentiated from shearing resistance and described in terms of arching action and mechanical arrangement of soil particles. Internal stability was defined as resistance to displacement arising from mutual support of adjacent particles in the mass. In quantitative terms, internal stability of a granular material was measured directly by the principal stress ratio and translated into terms of an angle of pressure transmission, θ, as in the following equation:

$$\frac{p_v}{p_h} = \cot^2\theta \quad\ldots\ldots\ldots\ldots\ (4)$$

Since 1936, there has been much work done and much interest in the testing of soil and soil mixtures under triaxial compression. The triaxial compression test has been adopted by many laboratories, both as a research tool and as a design test. Triaxial testing is still under development, and both the test procedure and interpretation of the test results are the subject of continued discussion (**10**). During this period, the Michigan laboratory has used the triaxial test largely for evaluating stabilized mixtures. It has rarely been used in connection with the design and construction projects of the nature under discussion in this paper, as the time and expense of obtaining the type and number of undisturbed samples required is usually too great.

However, such samples were obtained in connection with the design of caissons for the Conners Creek power plant of the Detroit Edison Co. This study serves to illustrate the relation between shearing resistance due to cohesion and internal stability and also provides an important link in the chain of development directed toward evaluating the bearing capacity of hardpan. It would appear that the most direct interpretation of the experimental results in Fig. 8 is expressed by the following equation, in which S_c is the shearing resistance due to cohesion:

$$p_v = \frac{2S_c}{\sin 2\theta} + p_h \cot^2\theta \ldots\ldots\ldots (5)$$

Analysis of these data by Eq 5 shows the shearing resistance due to cohesion to be 5110 psf and the angle of pressure transmission, θ, was 22 deg 38 min, computed as follows:

$$\cot^2\theta = \frac{330 - 100}{40 - 0} = 5.75 \quad \theta = 22 \text{ deg } 38 \text{ min}$$

$$S_c = \frac{p_v}{2} \sin 2\theta = \frac{100}{2} \times \sin 45 \text{ deg } 16 \text{ min}$$

$$= 35.5 \text{ psi} = 5110 \text{ psf}$$

On this project, a relatively small number (21) of Michigan standard core samples ($1\frac{3}{8}$ in. diameter) were taken from the preliminary test caissons for laboratory testing, as well as to obtain the penetration index correlation shown in Fig. 5. In these preliminary tests, shearing resistance, S_c, from the ring shear test varied from 1300 psf at the top of hardpan to 5000 psf in the densest hardpan at greater depth. The compression shear from the unconfined test varied from 2220 psf at the top of hardpan to 3165 psf at greater depth. During construction, 92 samples were taken from the bottom of the caissons at elevations close to the top of hardpan. On these samples, the average shearing resistance, S_c, from ring shear was 1295 psf, and the compression shear, 1925 psf. These data are cited particularly as they provide an example of another method of evaluating the angle of pressure transmission, θ, when triaxial compression tests cannot be run.

In connection with Fig. 8, the cohesion intercept, $\frac{2S_c}{\sin 2\theta}$, was used to determine the shearing resistance due to cohesion. This same relationship may be used in evaluating the angle θ from the ratio of unconfined compression to direct shear shown in Fig. 7. In so doing, the validity of the result will depend, first, on having a large enough number of tests to yield a representative average. Furthermore, it would be preferable to use the probability curve analysis, but the figures in the preceding paragraph are arithmetic averages which give a less satisfactory measure, but still a reasonable approximation.

In the second place, this interpretation involves some basic principles about which there appears to be considerable confusion. In an element of mass subjected to principal pressures such as shown in Fig. 8, the shearing resistance due to cohesion is mobilized on a plane steeper than the 45 deg plane of maximum shear. This means that the plane of failure is dictated by the grain structure or mechanical arrangement of the particles, which prevent failure on the plane of maximum shearing stress. The failure takes place on this steeper plane, thus increasing the maximum pressure difference by a mechanical advantage factor, represented by a value of sin 2 θ

less than unity. This is the contribution of mechanical stability and it must be so interpreted in order to preserve the integrity of shearing resistance due to cohesion as being independent of normal pressure and shearing stress, both of which vary with the angle θ.

It has become common practice to report the results of unconfined compression tests as one-half the observed compressive strength or shearing resistance, S_{uc}, further modified by the 4 to 1 ratio, to give an equivalent static shearing resistace, $\frac{S_{uc}}{4}$. As long as the material is purely cohesive and the angle of shear failure is 45 deg, $\frac{S_{uc}}{4}$ is numerically equal to S_c from the ring shear test. When the angle of failure, θ, is less than 45 deg, the following relations hold:

$$\frac{S_{uc}}{4} = \frac{S_c}{\sin 2\theta}$$

$$\sin 2\theta = \frac{4S_c}{S_{uc}}$$

From the data given above from laboratory shear test on hardpan samples from the Conners Creek plant, the angle of failure or angle of pressure transmission, θ, is 21 deg 14 min as compared to 22 deg 38 min from the triaxial compression tests. In the case of the City-County Building, the shear ratio was 5.0, instead of 4, as shown by the most probable value in Fig. 7. In this case, the angle θ would be:

$$\sin 2\theta = \frac{4}{5.0} = 0.80 \text{ deg } \theta = 26 \text{ deg } 34 \text{ min.}$$

BEARING CAPACITY OF HARDPAN

In order to bring field and laboratory determinations of soil resistance together in an integrated expression for estimating the bearing capacity of hardpan, a formula was presented in 1936 for the bearing capacity of granular materials, based on the definition of internal stability already cited in this discussion (8). This formula was extended to include the effect of cohesion and surcharge as given in the following equation:

$$q = \frac{1}{3} K w_1 r \cot^5\theta + \frac{2S_c}{\sin 2\theta}(1 + \cot^2\theta) + w_o h_o \cot^4\theta \quad \ldots\ldots\ldots\ldots (6)$$

Equation 6 has been used to make design estimates of the bearing capacity of hardpan based primarily on penetration measurements but supplemented by laboratory shear tests in a limited number of cases. On three major projects, full scale loading tests have been made on bearing piles, and they provide the final basis of correlation.

The first term in Eq 6 can be designated as internal stability. It contains one new factor, an edge coefficient, K, which was used to represent the resistance to displacement arising from pressure transmitted from the bearing area to supporting elements outside the central column, thus adding to the resisting lateral pressure. It was first introduced in the analysis of Berry's plate loading tests performed in the laboratory on five types of granular material, including four natural gradations of a sand and gravel and lead shot (8). K varied from 11 to 16 for the natural granular materials and was close to unity for the lead shot. Later, Davis (11) found a similar increase in bearing capacity and reported a range of experimental values of the same order of magnitude. Also, McLeod (12) included this factor in the bearing capacity of bituminous paving mixtures and suggested values of K ranging from 1 to 10.

The second term in the equation evaluates the integrated effect of cohesion in bearing capacity. Special note may be made of the fact that the principal stress difference on the element of mass from which lateral thrust is supplied is multiplied by the mechanical advantage factor, $\cot^2 \theta$, when it is transmitted to the bearing area through the granular structure.

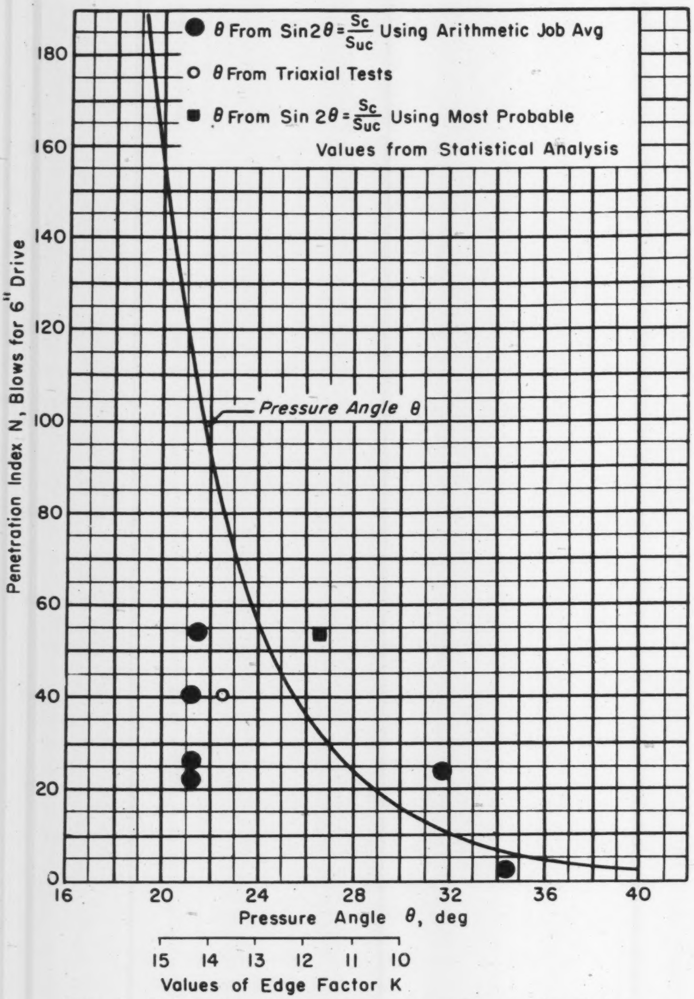

FIG. 9.—Internal Stability Factors *versus* Penetration Index.

The third term, which evaluates the effect of surcharge, needs no particular explanation as it contains only conventional factors of resistance. As a practical matter, the entire effect of surcharge magnified by a large mechanical advantage can only be mobilized if the supporting hardpan is virtually incompressible. This point has an important bearing on the magnitude of developed capacity as measured by field loading tests.

There are two factors of mechanical stability in Eq 6 that reflect the physical properties of the hardpan which must be determined quantitatively. These are the angle of pressure transmission, θ, and the edge coefficient, K. In general terms, it has been shown that both K and θ are affected most by the relative density and particle shape of the particular aggregate. In approximate terms, K may vary from 1 to 25 with the usual range in natural granular materials being from 10 to 20, increasing as the density increases. The usual range of variation in θ is from 30 to 20 deg, measured from the axis of maximum principal pressure, with the angle decreasing as the density increases.

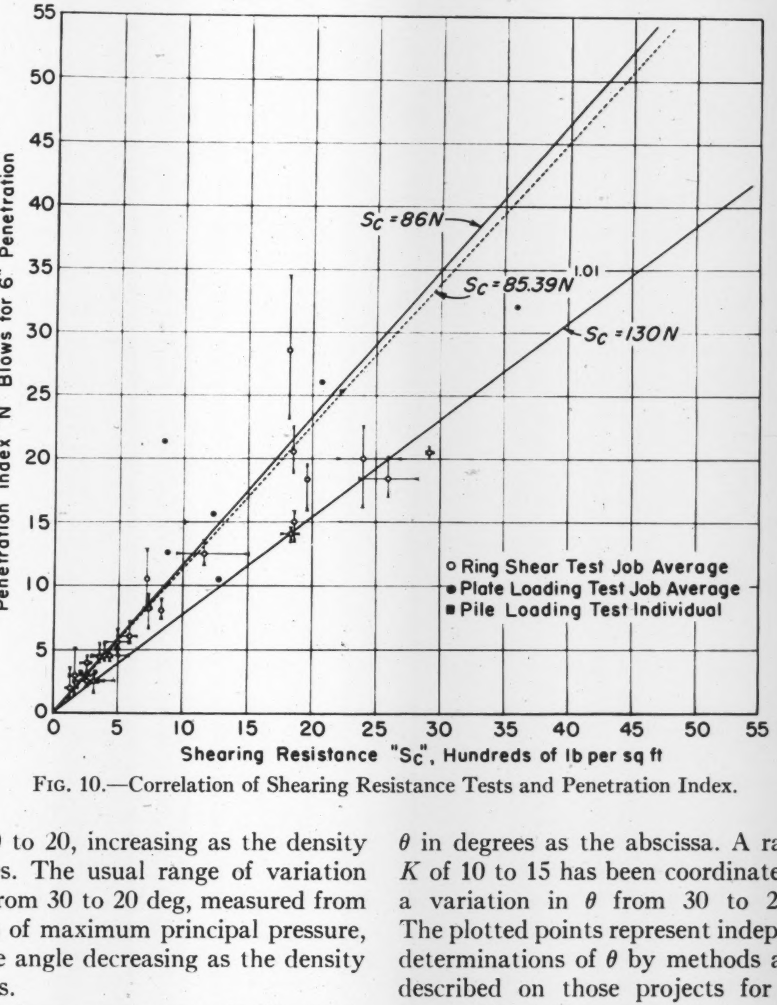

FIG. 10.—Correlation of Shearing Resistance Tests and Penetration Index.

With these ranges as a background, estimating K and θ was a cut-and-try procedure on each project where estimates of bearing capacity were required in preliminary design. The accuracy of this preliminary estimate was then checked by the field loading tests. From this accumulated experience, influenced by some theoretical considerations, the empirical relations shown in Fig. 9 have now been formulated. The curve itself follows the variation of $\cot^5 \theta$, with the penetration index as the ordinate and θ in degrees as the abscissa. A range in K of 10 to 15 has been coordinated with a variation in θ from 30 to 20 deg. The plotted points represent independent determinations of θ by methods already described on those projects for which penetration measurements were available.

The validity of the estimates of K can only be established by the results of pile loading tests, as there is no other measure available. However, other measures of θ are available, and the plotted points indicate that the estimate of θ in Fig. 9 is reasonable. There are still a relatively small number of check points, as these values are job averages on projects of considerable magnitude, and not too frequent in occurrence.

Correlation of Shearing Resistance and Penetration Index:

The other soil resistance factor of importance in Eq 6 is shearing resistance due to cohesion. Correlation of shearing resistance with the penetration index has been presented in Fig. 10 for values of direct shear from the ring shear test. In order to summarize the large number of individual test results available, points have been plotted only for job averages, with the range of shear values and penetration index being indicated by the length of the arrows. The data shown are from projects in which the penetration index was being measured with the Michigan penetrometer. Field penetration as measured by the Raymond standard has not been correlated except for the observations on hardpan at depths of 100 to 120 ft. Statistical studies are now in progress by which it is hoped to establish the variation of the Raymond penetration with depth below the surface in terms of shearing resistance from laboratory tests.

Other data shown represent shear values determined by field loading tests, some of which have been previously reported (1,2). The shearing resistance values from plate loading tests have been deduced from developed pressure in the linear equation for bearing capacity (1). The shear values from loading tests on friction piles involve the analysis of such tests which are outlined in the following discussion of field loading tests on piles.

Other corroborative data showing the correlation between penetration observations and other methods of measuring soil bearing capacity have been reported by McLeod (13). He conducted a comprehensive series of observations with parallel tests on clay subgrades for airport runways, including plate bearing tests, California Bearing Ratio (CBR), cone bearing (North Dakota Cone Test), Housel penetrometer, and triaxial compression tests. In summarizing the results of these tests on eight airports in Canada, he concludes that cone bearing and Housel penetrometer give the most consistent correlation with measured bearing capacity, and both are rapid tests involving only simple apparatus. In comparing the relative merits of the different types of penetration devices, it appears that there is little to choose between cones, driving points, or core barrels. The accuracy and reliability of any of the methods is more a function of precise measurement and logical analysis than of the type of penetrometer. The advantages offered by the use of the core barrel or sampling device is that it combines two objectives in one operation, that of obtaining the sample required for laboratory testing while making the penetration observation. The practical significance of this is that the penetration measurement can then be obtained in the normal process of sampling used in commercial boring practice. The result is valuable data at little increase in cost, and a considerable saving in time. This makes it possible to adopt more adequate soil investigation on a wider range of construction projects.

PILE LOADING TESTS

The simplest type of test for measuring shearing resistance has been a loading test on an open end steel cylinder pile. With such a pile, the friction or shear on the core and point resistance could be eliminated in some of the tests by removing the core. A particularly complete series of such tests was made by the Detroit Edison Co. at the Delray power house in 1931, and some of the results of these tests have been reported (14). Other structural steel shapes have also been used, and tests have likewise been made on timber and concrete piles. Altogether, there have been more than 300 field loading tests conducted to date on all types of piles, but correlation of side shear values obtained in later years with the penetration index is again awaiting completion of statistical studies of the Raymond penetration observations.

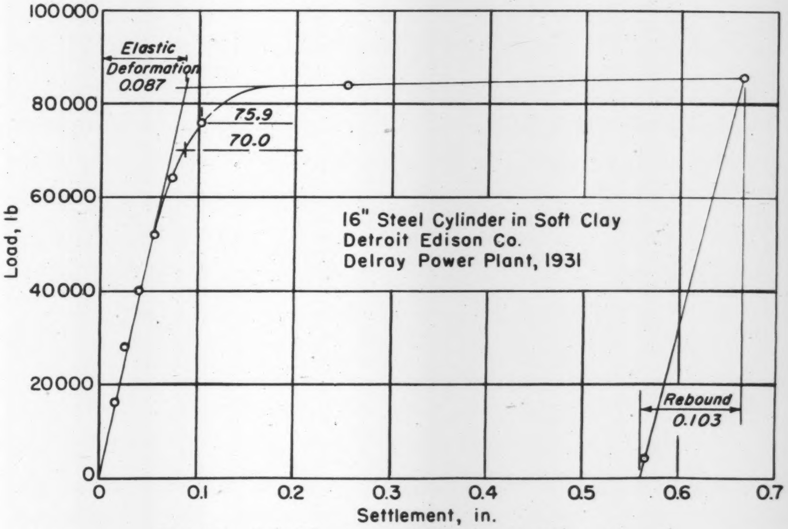

FIG. 11.—Load-Settlement Diagram for Shear Pile.

Loading Test Procedures:

The loading test procedure that has been evolved for piles follows in principle that used for laboratory shear tests and plate loading tests for soil bearing. The principle is that the strength of any material can be determined from its elastic properties and its ability to carry load without permanent deformation upon removal of the load, or without progressive yielding under load. With respect to permanent deformation, this principle has been enunciated by Reiner (15) as follows:

> "When a body has been transformed by being subjected to load . . . and the loads are removed, part of the deformation will be recovered. This part is elastic and is called strain. From this definition there follows that we cannot determine strain from a loading diagram in which the total deformation is recorded, but only upon unloading when we see how much of it is recovered."

In addition to determining the elastic limit or rebound, there is a second technique which is equally effective. This method is to determine the yield value of the supporting medium by measuring the rate of deformation for various loads and, by extrapolation, establish the load at which yielding or progressive deformation begins. This determination involves increment loading at constant time intervals from which consistent time *versus* rate of deformation relations can be obtained to establish the yield value.

This method has been accepted as an alternate procedure by the society in its Tentative Method D 1143.[4]

A third procedure which has been used is repetitive loading. This is an effective means of establishing the character of the permanent deformation or set, however small, which takes place upon the application and removal of load in each loading cycle. This gets into the field of fatigue testing and, while it is time consuming and expensive on the scale of field loading tests, the results are often so illuminating that it is justified in certain cases.

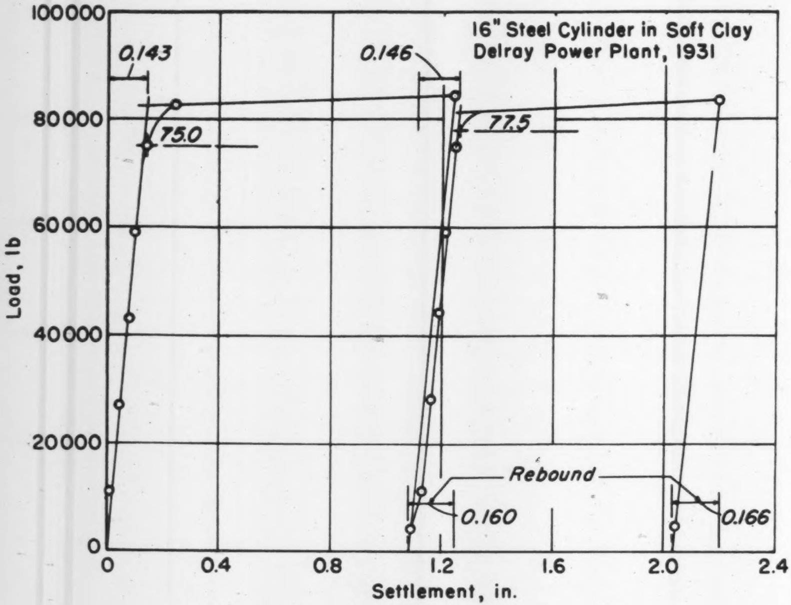

FIG. 12.—Repetitive Loading Cycles on Shear Pile.

All three of these methods have been incorporated in typical specifications for conducting pile loading tests which follow, the essential parts of which have been incorporated in the new Detroit Building Code (**16**).

The ultimate capacity of the pile shall be defined in these tests by any one or the average of the following determinations, whichever is judged by the engineer to be the most representative under existing conditions.

1. The yield value, or load at which progressive settlement begins, as determined by extrapolation of the rates of settlement for each load increment.
2. The load carried at a settlement equal to the measured elastic deformation or rebound.
3. The repetitive load at which there is no further permanent deformation, or at which the rebound approaches 100 per cent of the measured settlement.

Repetitive loading cycles are normally required only under conditions in which the determinations under (1) and (2) are inadequate.

Typical Results of Pile Loading Tests:

Data to support the validity of the test procedures outlined above and the determinations of ultimate supporting

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[4] Tentative Method of Test for Load-Settlement Relationship for Individual Piles (D 1143 – 50 T), 1955 Book of ASTM Standards, Part 3, p. 1812.

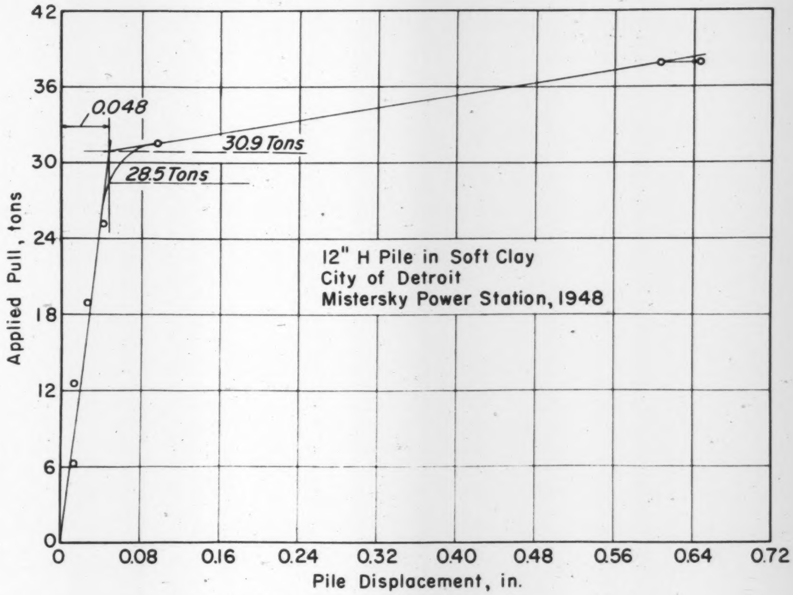

FIG. 13.—Pull-Displacement Diagram for Shear Pile.

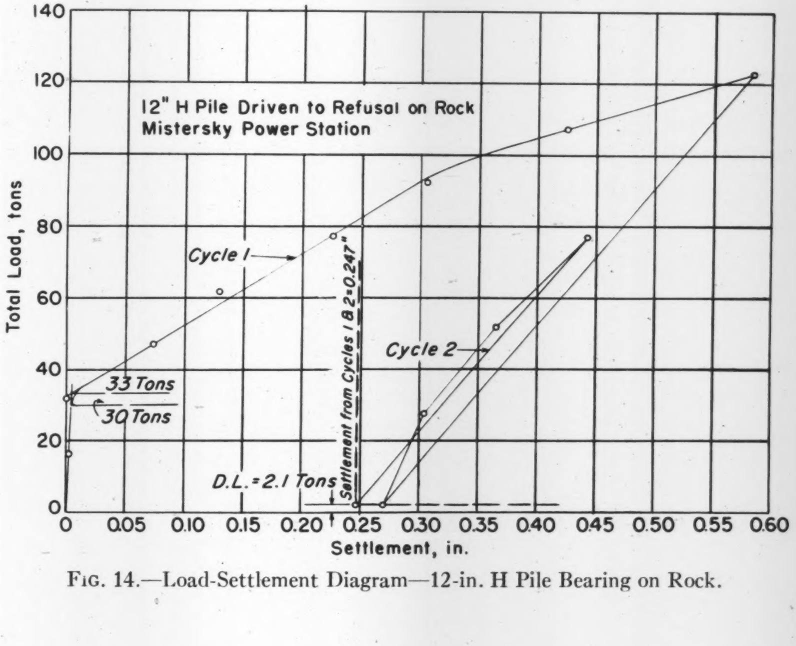

FIG. 14.—Load-Settlement Diagram—12-in. H Pile Bearing on Rock.

capacity are presented in a series of tests selected from typical projects. The results as shown on the various diagrams stand on their own feet, and the review and comments will be as brief as possible. The tests include some typical tests on friction or shear piles to cover the measurement of side shear. This is a factor even in bearing piles, although they deliver the major part of the load to some highly resistant substratum such as hardpan or rock.

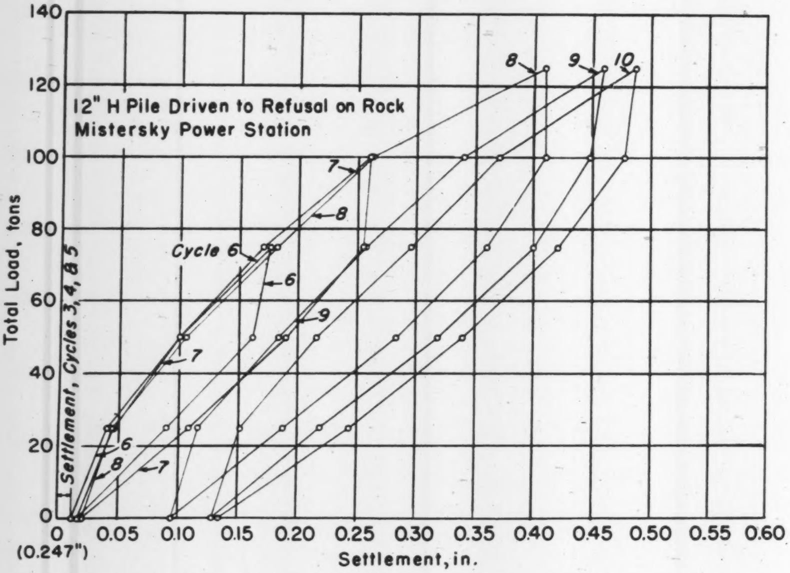

FIG. 15.—Repetitive Loading Cycles—12 in. H Pile Bearing on Rock

Figures 11 and 12 illustrate the behavior characteristics of a pile supported almost entirely by side shear in a purely cohesive clay. In all cases, there is an extended range of elastic behavior with settlement proportional to the load. This is followed by a rather abrupt transition to progressive settlement with negligible increase in load, and finally a rebound equal to or slightly greater than the elastic deformation determined by intersection of the tangents to the load-settlement diagram. The rebound is a measure of the elastic limit of the supporting medium after such improvement as may have resulted from the loading cycle. The clay involved in these two tests is soft and plastic and may be classified as a saturated clay. The distinctive behavior of these piles is further marked in Fig. 12 by the evidence that the permanent settlement is all shearing displacement rather than consolidation and the fact that repetitive overloading under static load neither impairs nor improves the supporting capacity.

Figures 13, 14, and 15 are all taken from the Mistersky power station project, in Detroit where soil conditions are similar to those at the Delray power plant, except that the clay was somewhat softer. The shearing resistance by the ring shear test varied from 50 to 150 psf with an average value of 110 psf for a depth of some 90 ft above hardpan. The validity of the shear tests was questioned, so several test piles were driven, the first one of which was stopped

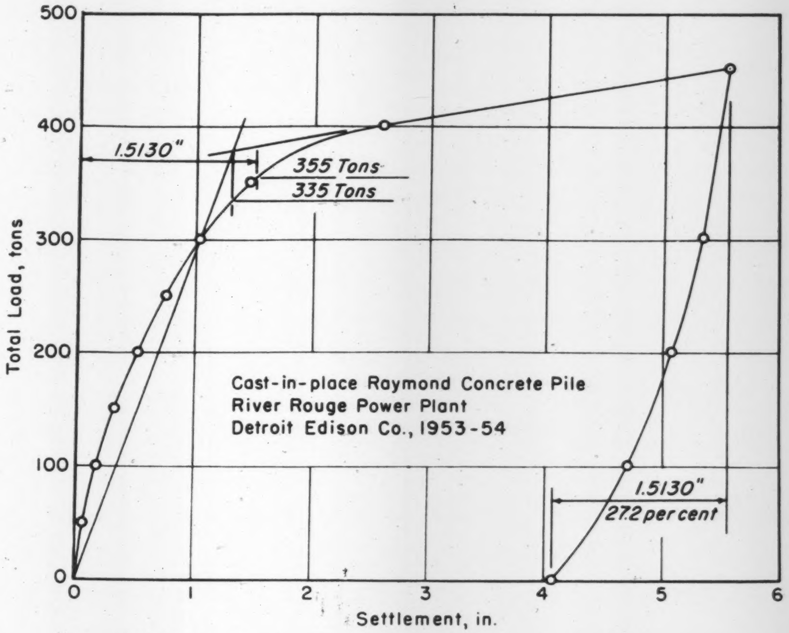

FIG. 16.—Load-Settlement Diagram—Pile Bearing on Hardpan.

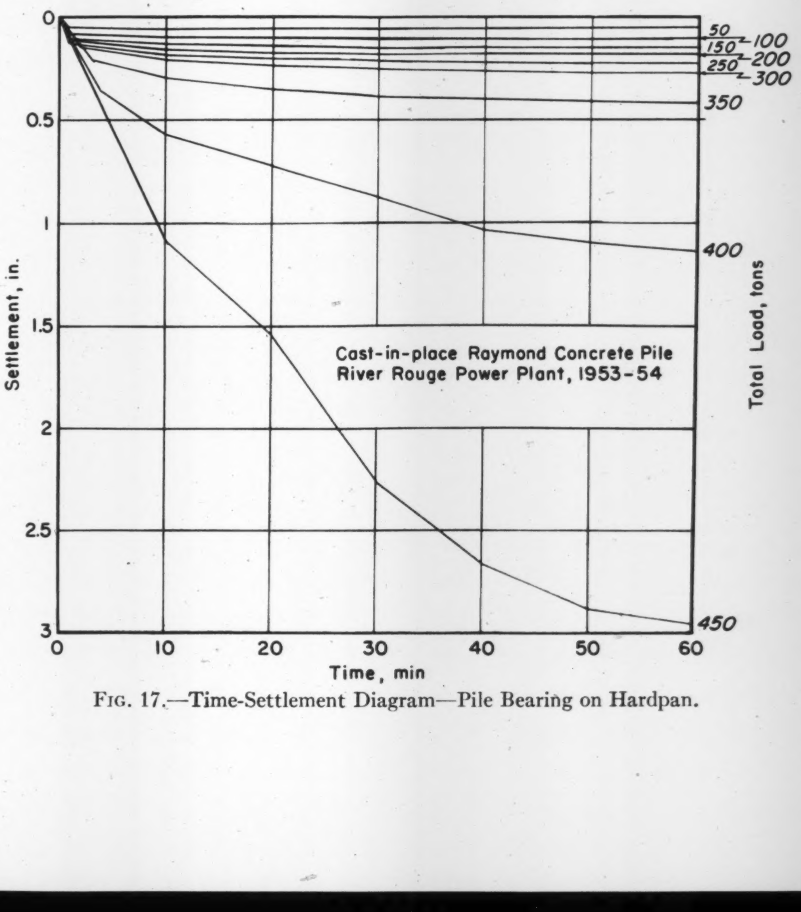

FIG. 17.—Time-Settlement Diagram—Pile Bearing on Hardpan.

short of hardpan and subjected to a pull test. These piles were 12-in. H piles, providing a shear area of 6 sq ft per ft of steel in contact with soil.

Prior to testing, the load required to pull the pile was computed from the shear tests as varying from 27.5 to 30 tons, depending upon whether the pile pulled clean or pulled with the flanges filled with clay. The results shown in Fig. 13 satisfied the questions that had been raised.

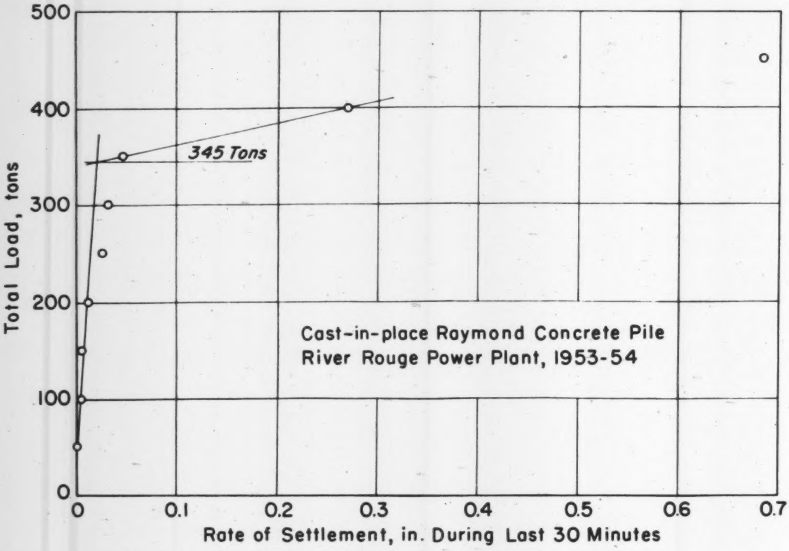

FIG. 18.—Yield Value Diagram—Pile Bearing on Hardpan.

Figures 14 and 15 show the results of a similar 12-in. H pile that had been driven through approximately 4 ft of hardpan to refusal on the underlying limestone. Figure 14 shows the first two loading cycles, and particular attention is directed to cycle 1. There is an abrupt change in slope of the load-settlement diagram just in excess of the 30 tons computed as the side shear above the hardpan. In this connection, it should be stated that there is no adequate basis for estimating the side shear in hardpan on the vertical steel surface of the pile after the hardpan had been disturbed by driving. The total side shear load actually developed exceeded that developed in the overlying clay by only a small amount.

It is much more important to note in cycle 1 that after the side shear had been exceeded, the 12-in. H pile knifed into the rock as shown by the considerable increase in total settlement and permanent deformation. While the amount of permanent settlement was only 0.25 in., there was positive evidence of increased penetration.

Following the first two loading cycles, the series of repetitive loads was continued as shown in Fig. 15 to explore the action of the H pile on rock. Cycles 3, 4, and 5 have not been plotted, but loads applied did not exceed 50 tons and the additional permanent settlement was very small. The same is true at loads of 75 and 100 tons in cycles 6 and 7. However, when the attempt was made to increase the load to 120 tons, additional penetration resulted. Three load repetitions at

120 tons demonstrated that the permanent settlement after each cycle was decreasing. The results indicate that permanent stability was being achieved, and it may be presumed that after a sufficient number of repetitions, the rebound would be 100 per cent.

Figures 16, 17, and 18 have been taken from a recent project to illustrate the typical behavior of high-capacity bearing piles designed and driven to mobilize the maximum supporting capacity available on hardpan. This particular pile was loaded to a maximum of 450 tons, and it has become common practice to test such piles up to maximum loads of 500 tons or more. These three figures also illustrate the testing procedure and methods of analysis for such pile loading tests that represent what is becoming standard practice in the Detroit area.

The load-settlement diagram in Fig. 16 provides two methods of establishing the elastic limit which may be defined as the load carried at the maximum elastic deformation. The elastic deformation may be taken as the intersection of tangents to the load-settlement diagram or equal to the total rebound. The rebound is considered a more reliable measure of the total capacity of the supporting medium as it includes the improvement due to the load application. Rebound and elastic deformation may approach equality after a sufficient number of loading cycles have been conducted to establish complete elastic behavior.

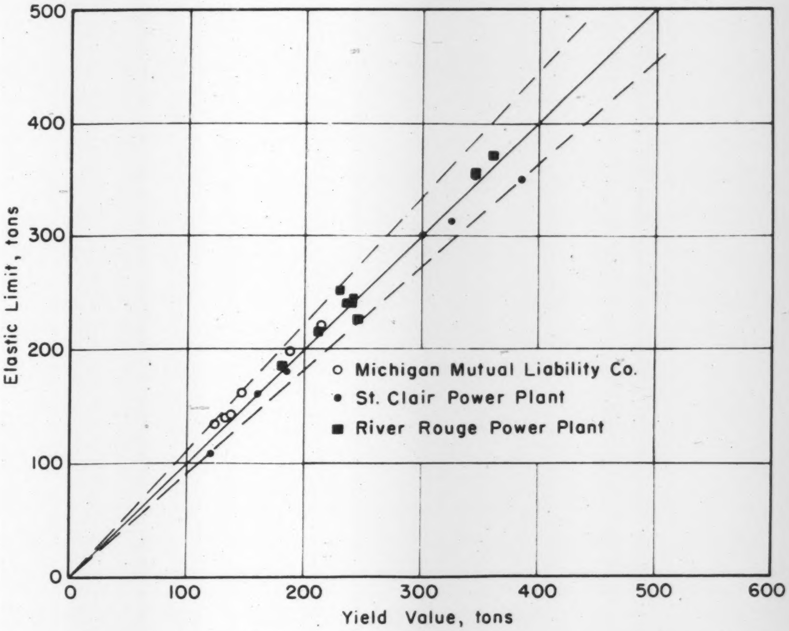

FIG. 19.—Comparison of Yield Value and Elastic Limit from Pile Loading Tests.

Figures 17 and 18 illustrate the second method of establishing maximum capacity of a bearing pile by determination of the yield value. Figure 17 shows a typical set of time-settlement curves from which a terminal rate of settlement may be established for each load increment. The spread of these curves indicates the beginning of yielding, but plotting their slopes against load as in Fig. 18 is a positive determination of the yield value.

It may be noted that the three separate determinations of the maximum capacity of the pile under discussion check within rather close limits. Close agreement between the elastic limit and the yield value is not unusual when the control of load application follows the precision technique outlined herein and the analysis of test results also follows precise methods. The close agreement between the elastic deformation and rebound only occurs when the supporting soil is completely consolidated. In many cases, this may not be true and the rebound alone must be accepted as the measure of the elastic limit. Figure 19 further illustrates the close agreement found between the load at the rebound elastic limit and the yield value. These data include all complete loading cycles on the four projects on which the bearing capacity of hardpan has been evaluated by pile loading tests.

TABLE I.—SUMMARY OF PHYSICAL PROPERTIES OF HARDPAN-CORRELATION OF ESTIMATED AND MEASURED VALUES.

Project[a]	Penetration Index, N		Resistance Factors			Radius of Area, r, ft	Static Head, h, ft	Computed Bearing Capacity				Measured Bearing Capacity, M, tons per sq ft	Ratio, $\frac{M}{C}$, per cent
	Range	Average	Shear Value, S_c, lb per sq ft	Pressure Angle, θ, deg	Edge Coef. K			Internal Stability, kips per sq ft	Cohesion, kips per sq ft	Surcharge kips per sq ft	Total C, tons per sq ft		
A.....	18 to 75	41.6	3580	25.6	12.2	0.564 0.795	108 108	13.3 18.7	50 50	265 265	164.2 166.9	68.7 51.1	42 31
B.....	19 to 80	42.6	3660	25.3	12.3	0.47 0.47	119 118	11.3 11.3	52 52	297 295	180 179	96.4 193 (*a*)	54 108
C.....	49 to 148	98.0	8430	21.8	14.1	0.47 0.687	106 106	29.8 43.6	175 175	525 525	364.9 371.8	261 236	72 64
D.....	30 to 106	54.5	4690	24.2	12.8	3.5	94	114.0	75	292	240	(25) (*b*)	(10+)
E.....	15 to 83	64.2	5520	23.5	13.2	0.50 0.583	80 80	19.7 23.9	95 95	280 280	197.3 199.4	244 282	124 141

[a] Project A—Conners Creek power plant—Detroit Edison Co.
Project B—Michigan Mutual building—(*a*) Driven to refusal.
Project C—St. Clair power plant—Detroit Edison Co.
Project D—City-County Building—(*b*) Maximum design pressure for caissons.
Project E—River Rouge power plant—Detroit Edison Co.

Computed bearing capacity = Internal stability + Cohesion + Surcharge

$$= \frac{Kw_2 r \cot^5 \theta}{3} + \frac{2Sc\ (1 + \cot^2 \theta)}{\sin 2\theta} + w_1 h \cot^4 \theta.$$

Density of clay overburden = w_1 = 125 lb per cu ft.
Density of hardpan = w_2 = 140 lb per cu ft.

There remains one final comparison that seems necessary to give a complete picture of the developments in the design of deep foundations in the Detroit area. This development may be best described by pointing out several significant trends which have become evident, illustrated by the numerical values summarized in Table I.

In this connection it is first interesting to note that estimates of bearing capacity by Eq 6 result in predicted bearing capacity of several hundred tons per square foot. These estimates assume full mobilization of shearing resistance due to cohesion, internal stability and surcharge, all magnified by the mechanical advantage of the granular structure. The predicted bearing capacity in order of magnitude is 8 to 10 times the nominal bearing values which may be found in standard handbooks and building codes for rock. The use of such high bearing values would be quite out of the question unless they could be demonstrated beyond any reasonable doubt by both theory and laboratory experiment, backed up by full scale loading tests. That is what makes the comparative figures in Table I of such interest.

Field penetration measurements on five major projects have been translated into the soil resistance factors, S_c, θ, and K. The bearing capacity of hardpan has then been estimated in terms of the three sources of resistance in Eq 6, designated as internal stability, cohesion, and surcharge. In four of these projects, the actual bearing capacity of the hardpan has been measured by field loading tests. In each case, the test loading has been reduced by the amount of side shear to obtain the net load transmitted to hardpan by bearing at the point of the pile. The side shear has been computed from shear values above hardpan from laboratory tests and estimated from the penetration index in hardpan.

In the last column is shown the percentage of the computed bearing capacity that has actually been mobilized in the field loading tests. In the case of one project, the City-County Building, there were no loading tests performed, and the maximum pressure used in the design of the caissons has been shown for comparison. With this one exception, it is interesting to note that the measured bearing capacity is of the same order of magnitude as that computed, that is, several hundred tons per square foot.

One of the most significant features of these data is the demonstration that, by heavy driving, it is possible to develop the maximum supporting capacity of hardpan exceeding, in some cases, the capacity computed from average penetration resistance. In project *A*, the loading tests were on bearing plates set at the bottom of a test caissons and no driving was involved. Analysis of tests indicated that the hardpan was not sufficiently incompressible to mobilize the surcharge at the full mechanical advantage of the granular structure. This may be contrasted with project *E*, at the River Rouge power plant, where unusually heavy driving was employed. The piles were driven through the less-resistant hardpan, and this, in combination with possible preconsolidation, resulted in the high capacity developed.

SUMMARY

The penetration method of measuring soil resistance first presented to the Society in 1935 has been extended to a wide variety of soil conditions in the solution of design and construction problems. The quantitative relationships which have been used are presented in the following summary:

1. The equivalent shearing resistance, S_c, from cohesion has been correlated with the University of Michigan penetration index, N, in terms of an impact factor, C_I.

$$S_c = 1.2\, C_I N \ldots\ldots\ldots\ldots(3)$$

2. The impact factor C_I, has been evaluated empirically and varies from as low as $\frac{1}{2}$ for plastic impact to values approaching unity representing elastic impact. The value of $\frac{1}{2}$ is used for commercial boring operations with considerable lengths of flexible drill rod or

pipe. Shearing resistance is then given by the following equation:

$$S_c = 86\,N \ldots\ldots\ldots\ldots (3a)$$

3. The Raymond Penetration, R, widely used in commercial boring practice has been correlated with the University of Michigan penetration index, N, for sampling in hardpan at depths of 100 to 120 ft. The following empirical relationship applies to that depth but for other lengths of drill rod or pipe this numerical factor has yet to be established.

$$N = 0.31\,R$$

4. The internal stability of granular mixtures has been expressed in terms of an angle of pressure transmission, θ, and a dimensional coefficient, K, included in the bearing capacity equation first presented to the Society in 1936 (**8**). Curves showing the variation in these two physical properties with the penetration index, N, are given in Fig. 9 and were used in estimating the bearing capacity of hardpan.

5. The estimates of shearing resistance based on penetration measurements are compared with independent laboratory shear tests and field loading tests including both plates and friction piles. The correlation of these data is shown graphically in Fig. 10.

6. Estimates of the bearing capacity of hardpan by the penetration method have been substantially confirmed by the results of full size field loading tests. The results of these tests are presented graphically in Figs. 16 to 19 and the correlation has been summarized in Table I.

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DISCUSSION

Mr. A. L. Bentley, Jr.[1] (*by letter*).—The author's paper constitutes a significant step in the control of deep caissons founded in hardpan. However, it is stated that experience with the subgrade penetrometer in connection with deep caisson construction is limited to only two major projects; therefore it is the purpose of this discussion to present some preliminary data compiled on a third major caisson project which is now under construction. This project consists of 51 bell and shaft type caissons carried to depths of approximately 103 to 110 ft which are landed in a very variable hardpan with respect to the amount of clay binder, in some cases having none at all. The caissons are to support a power house addition of the Toledo Edison Co., and its program for their control was instituted with the cooperation of the Toledo Edison Co. and the assistance of the University of Michigan and the University of Toledo.

At first, the only form of control consisted of converting the standard penetration test results obtained by the Raymond Concrete Pile Co., during the test boring program for this project, to values of the Michigan penetration index. These index values were utilized to compute ultimate bearing capacity of the hardpan from Mr. Housel's 1936 formula for bearing capacity of granular materials. All the variables in that formula are functions of penetration index or easily estimated units, leaving the diameter of the bell as the design variable. For these bells, the computed ultimate bearing capacity is enormous; therefore, in an effort to reduce its values to the present general conception of this material they were reduced by a factor of 10 to a value called presumptive ultimate bearing capacity, and factors of safety were computed therefrom. The depth of the caisson and the bell diameter were computed from the converted Raymond penetration, and the material in the bell was checked visually with the samples submitted. This is the essence of the control program as originally intended.

The appearance of this paper produces the addition of the use of the subgrade penetrometer to the control program. It is utilized to extract record samples and also samples for unconfined compression and transverse shear tests in an effort to check the validity of the design method. It is also utilized to serve as a check on the converted Raymond penetration values at the bell elevations in order to compare local conditions with the average of the widely scattered test borings, in view of the inconsistencies in the quality of the hardpan.

Several interesting observations may be made at present, even on the basis of preliminary data. The highly granular character of the hardpan has produced highly scattered results of the transverse shear tests, so scattered as to make them useless. The results of the unconfined compression tests on the hardpan have shown virtually a 330 per cent increase in resistance to penetration over that produced by cohesion alone. This is due almost entirely to the frictional resistance of the highly consolidated granular material. The presence of stone and the variations of weight of the subgrade penetrometer preclude the use of Fig. 3 in computation of penetration index. The use of the penetrometer has virtually eliminated the cumbersome deep well load test from consideration as an element of caisson control, except as a check on the validity of the penetration analysis.

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Thus far, on the basis of the preliminary data, no determination of the ratio of compression shear to transverse shear has been directly possible due to the erratic results of the transverse shear tests on the hardpan. Indirect analysis of the results of penetration tests performed in hard, sandy clay at higher elevations indicate a ratio of 5. This clay has the same level of compression shear but only 22 per cent of the penetration resistance of the hardpan. This is due to an estimated rise of angle of internal friction from 15 deg in the clay to a value near 44 deg in the hardpan. The element of localized control by use of the subgrade penetrometer depends upon the accuracy with which it reflects the test borings in resistance to penetration. A comparison between the penetration index of the hardpan as taken by the penetrometer in the caissons to the

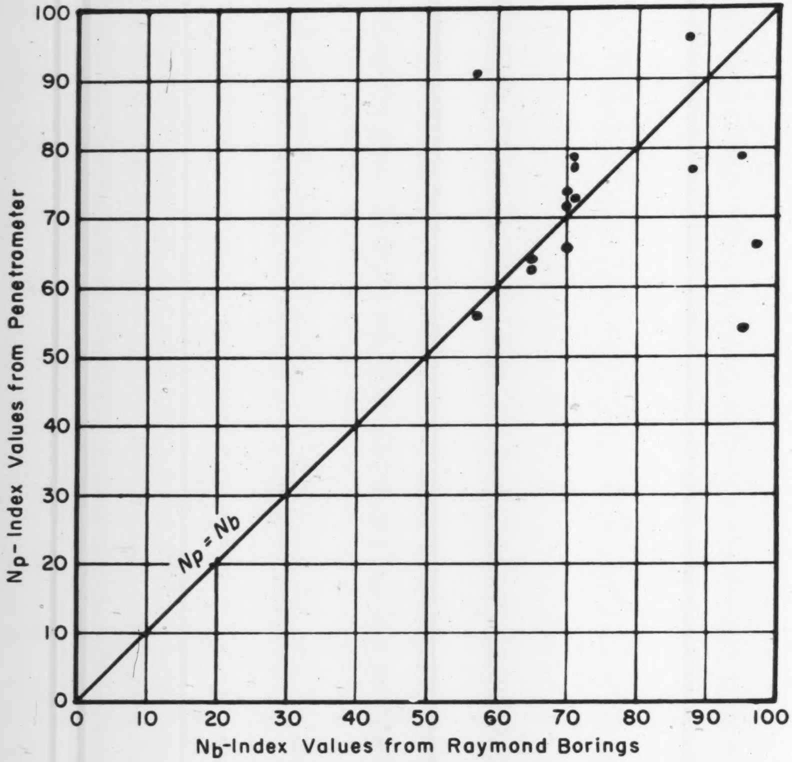

FIG. 20.—Preliminary Field Observations of Penetration Data.

average penetration index computed from the Raymond values for design purposes is shown in Fig. 20. This chart is based on only sixteen determinations of resistance; however, it indicates a reasonable degree of correlation similar to that in Fig. 5 of the paper. The Raymond penetrations reported on this project were for lengths of drive less than 12 in. due to the high resistance of the hardpan; therefore, the penetration index had to be computed from the factors shown in Fig. 4 and allowing for an impact correction of 90 per cent. They were then plotted *versus* depth, and a selected average arrived at for design purposes. The local penetrations in each bell were plotted against the selected boring value in Fig. 20. The scatter in Fig. 20 seems due to a difference in compressibility of the hardpan in each case, since the element of mechanical advantage due to overburden is absent in the case of the subgrade penetrometer but is present in the case of the test boring resistance. This element of total bearing capacity cannot be mobilized in the case of slight compressibility.

The local shear conditions reflected by the unconfined compression test in each caisson will serve to reevaluate the cohesion factor in Mr. Housel's 1936 formula. The term S_c was made equal to $86N$ for design purposes on the basis of the Raymond borings but will be changed to a constant value based on the average compression shear value divided by five. This new value of S_c will be only about 25 per cent of the original estimate.

In short, the penetration method has brought a high degree of field control to the design and construction of deep foundations of the caisson type.

MR. W. S. HOUSEL (*author's closure*).—The author was glad that Mr. Bentley could take the time to report his experience with the penetration method as outlined in the original paper. Opportunities to test the validity of field observations and design procedures under practical working conditions are all too few and none should be lost. That Mr. Bentley, working quite independently, was able to utilize these procedures and use them to advantage in the design of caissons for the Toledo Edison Co. plant is a source of gratification to the author.

There are several points in Mr. Bentley's discussion upon which some comment may be made. It is noted that the penetration method had only been used for design of caissons on two major projects. This is correct but it should be emphasized that the major source of checking the validity of the field observations and design procedures has been field loading tests on bearing piles. Four additional projects are cited in this connection and there were several other projects in progress at the time the paper was written which have since been completed.

Mr. Bentley notes that the bearing capacity computed from penetration in highly consolidated granular material is enormous and he applies a reduction factor of 10 to obtain a presumptive ultimate bearing capacity more in line with conventional practice. Such a reduction is understandable and provides a high safety factor quite desirable in deep foundations. Settlement is also a contingent factor of great importance and in hardpan not completely consolidated may justify a large reduction in capacity allowed in design. However, these practical considerations should not lead to overlooking the fact that this apparently enormous ultimate bearing capacity was actually developed in the field loading tests on piles. In the case of bearing piles, heavy driving may be used to preconsolidate less dense hardpan or drive through the weaker strata. Particular reference in this connection is made to project *E* in Table I where measured bearing capacity considerably exceeded that estimated. This was the result of heavy driving and consolidation due to loading.

There is one other comment that may be made and that has to do with the statement in the discussion regarding the high frictional resistance of the consolidated hardpan. Thinking of the resistance to penetration or displacement as a function of shearing resistance due to internal friction is the conventional concept. The author would like to point out that the resistance of granular materials to displacement is conceived in this paper as internal stability due to mutual support between particles and quite different from internal friction as it is universally defined. This is basic in the treatment of granular materials on which this development of bearing capacity relations is based.

APPLICATIONS OF ENVIRONMENTAL TESTING OF SOILS*

By Donald M. Burmister[1]

Synopsis

This paper is intended to illustrate the practical applications of environment testing of soils, in order to obtain more representative, adequate, and reliable information and soil test data for use in practical problems in soil and foundation engineering. The problems discussed show what environmental conditions and conditions imposed by the construction of structures should be taken into account in the testing and how they may be taken into account in the soil testing in order to achieve the above objectives. Standards of soil testing are used as the basic framework for the testing, but in each case special provisions and modifications are introduced in order to fit the testing procedures and techniques to the special environmental conditions that control. The principal objective in the soil testing is faithfully to reproduce the environmental conditions and imposed conditions that control in each situation in accordance with simulated service testing principles, which are becoming more and more commonly used in testing manufactured products in industry. This is learning how to test and to treat real soils under essentially real conditions, as an important forward step in soil testing procedures and techniques.

Soil testing is a most essential and basic aspect of soil investigations for foundations of structures and earthworks. It is really a work of discovery to disclose, to bracket properly, and to take fully into account the kind and range of soil character and the environmental influences that control responses. The major objective is to obtain representative, reliable, and adequate soil test data for use in practical problems in soil mechanics and foundation engineering. The problems cited from a large number of investigations illustrate and show what natural environmental and imposed conditions should be taken into account in soil testing, and to "spell out" in some detail how representative character and responses of soils can be consistently determined thereby.

Three fundamental concepts (**1**)[2] should govern attitudes, thinking, and practices in testing soils, which are the inherently variable and complex products of geological and weathering processes and of the natural environment.

1. The character and responses of soils are predetermined and governed by the influences of environmental conditions. Soil, due to the nature of its character and responses, is strongly susceptible to the influences not only of the natural environment but also of the

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1371.

artificial environment imposed in a soil test method.

2. Soil test procedures should adequately and representatively reflect the influences of the environmental and imposed conditions. The basic problem of testing soils is that of reconciling natural environmental conditions and the artificial environment imposed in a soil test. If test conditions are not representative of a situation, then the measured responses of the soils are certain to be modified from the original responses accordingly, but in an unknown manner and degree. Hence the test data will be deficient in these essential aspects. In order to insure adequacy of soil test results, it is necessary to learn what the conception of being representative really means in each method of test, when applied to specific conditions, and how it may be attained practically and consistently in soil testing.

3. The controlling influences and complex interaction of environmental and imposed conditions on soil responses can be determined reliably and adequately only by soil tests that are representative of and essentially reproduce the influences of these conditions prevailing or to be imposed under field conditions. The major emphasis should be given to the concept of adequacy of test results, because this is determinative in their interpretations and uses, and in the real adequacy of design of foundations and earthworks based thereon.

PRINCIPLES OF ENVIRONMENTAL TESTING OF SOILS

The problems of soil testing are principally those of "Controlled Environment Testing of Soils" (1) to bracket and to evaluate representatively the variable and complex conditions encountered in practice. Standards of controlled environment test methods can provide a basic framework of principles of soil testing for each method of test. A test procedure can be made to reproduce and to reflect significantly and representatively specific environmental conditions that control through special provisions and alternate test conditions and procedures within the basic framework. The real test for selecting and modifying alternate test conditions is that of making them representative, as the valid and practical approach.

In many cases these modifications of test conditions may be relatively simple and not involved, but they will be found to be the common sense approach. However, the problems of making strength tests of soils representative are inherently more difficult and involved, requiring technical skill and understanding, but the attainment is also much more important.

IDENTIFICATION TESTS

Applications of these concepts and principles are made first of all for the identification tests, such as sieve analyses, hydrometer tests, liquid and plastic limits tests, and laboratory and field identifications of soils by visual and manual methods. Here is the first opportunity in each situation for discovering the essential features of the real soil character, particularly with regard to their favorable and unfavorable aspects and their suceptibility to environmental influences, or to possible methods of improvement of certain significant qualities (2, 3).

Case 1:

This case concerns certain aspects of an investigation for the installation of a large storm drain adjacent to a railroad bridge abutment, requiring unwatering of the site and underpinning of the abutment without interruption of railroad traffic. Some 23 quart jars of samples obtained from 3 borings by a standard 2-in. by $1\frac{3}{8}$-in. split barrel sampler were tested by a soil laboratory to determine their grain size characteristics. A part of each sample was dried and tested by sieving with a final washing through the No. 200 sieve. No significant information was supplied on the character of the material passing the No. 200 sieve.

The author was called in later to evaluate the test data with regard to the drainage characteristics of the soils for the unwatering of the site by wellpoints, and with regard to the adequacy of the bearing values of the soils beneath the abutment for the underpinning operations. It was found to be impossible to evaluate these data sufficiently reliably for these essential purposes. It was considered essential either to obtain the remainder of the original samples, if available for examination, or to request additional borings. Fortunately, the unused portions of the samples were still available, as well as the dried sieve fractions. It was immediately evident, from an examination of the two bottles for each sample, that a completely unrepresentative specimen had been taken for the sieve analyses in every case. The sieve specimens were about 60 per cent of the total sample (dry weight basis) but they contained practically all of the gravel larger than the No. 4 sieve from the entire sample.

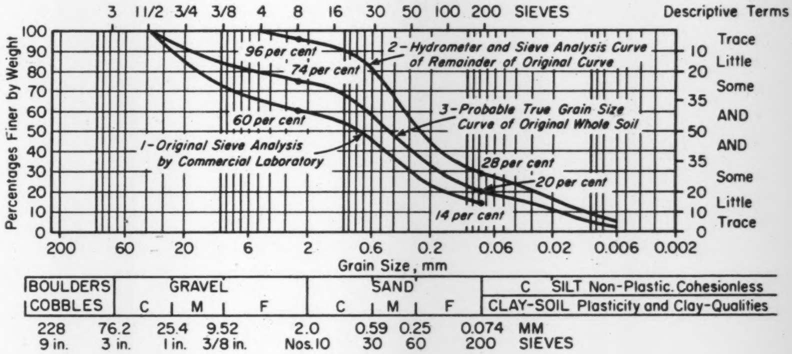


FIG. 1.—*Case 1.*—Evaluation of Probable True Identification and Grain Size Curve of Soil Sample Unrepresentatively Tested.

Probable true identification—Brown coarse to fine SAND, little Clayey Silt, some medium to fine Gravel. Slight PI.

Original sieve analysis specimen—	301 g dry weight	61.4 per cent
Remainder of original sample—	189 g dry weight	38.6 per cent
	490 g	

Relation for estimating probable true percentages for any grain size, D

Percentage finer by weight (Curve 3) = per cent (Curve 1) × 0.614 + per cent (Curve 2) × 0.386

The soil from each remaining moist specimen was identified by visual and manual methods (**3**, pp. 428–433) to determine whether silt or clay dominated the character of the soil. There was not enough material to make both grain size analyses and consistency limit tests. The identifications revealed that the soils were quite clayey and plastic in character. The evaluation of the data then required making grain size analyses of the remaining moist soil of all samples. It was possible to make a combined sieve and hydrometer analysis by the vertical stirring rod method for the dispersion of these specimens containing such large quantities of sand, allowing practically all of the sand to settle out of suspension in the last 20 sec of the final dispersion. A sieve analysis was performed after the hydrometer test. The grain size curves of the original sieve specimen and of the remaining specimen, together with identifications of the materials and other pertinent data are given in Fig. 1. for a typical sample.

The probable true grain size curve of the original soil sample was evaluated by taking proper proportions, as indicated in Fig. 1 of the two curves, for a sufficient number of particle sizes to define the new grain size curve. The probable original soil contained considerably less gravel, but more material passing the No. 200 sieve of a clayey silt character. The drainage characteristics (**9**, pp. 14–15, 23) of the probable original soils are therefore less favorable than might be anticipated from the original sieve data, and near the lower limit of successful well point dewatering in the upper stratum, but were somewhat better in the underlying stratum. The potential supporting values (**4**, Fig. 4) of the soils beneath the abutment were revealed to be poorer than would be indicated by the relatively higher gravel content and lower per cent of material passing the No. 200 sieve of the original sieve data. But there was now a greater consistency of relations between soil character and the driving resistances of the sampler.

The testing had thus obscured these relatively unfavorable aspects and was unrepresentative and deficient. It should be clear that no soil testing should be undertaken without essential information or knowledge being obtained or supplied on the nature of the problems involved and of the conditions that control.

Case 2:

This case concerns the investigation of borrow material for an extensive railroad grade crossing elimination involving some 300,000 cu yd of embankment material. Grain size analyses of borrow materials were made by a soil laboratory by sieving with a final washing through the No. 200 sieve. Consistency limit tests were made to indicate the character of the material. No data on the amount of oversize material was supplied. The author was called in later to establish criteria for compaction and construction control and to prepare specifications for construction. There was a limitation fixed by the railroad's practice on the amount of material passing the No. 200 sieve of 25 per cent for acceptability of borrow material for embankments. The grain size analyses submitted were of very little help and were deficient for these purposes, particularly for determining whether the limitation of 25 per cent passing the No. 200 sieve was either adequate or justifiable in this case.

In the most favorably located borrow area on a side-hill close to the embankment site large bag samples were obtained from three bulldozer cuts at depths of $1\frac{1}{2}$ to 4 ft and 5 to 7 ft, which provided a reasonably representative coverage for these purposes. The soil was the product of extensive weathering of diabase under temperate humid climatic conditions. It was an orange-red color containing partially weathered fragments of the dark gray original rock. This color indicated considerable oxidation of ferrous iron to the ferric state in the soil. The samples were taken in the spring of the year with temperatures below freezing and with the soil in a very wet condition. Construction was scheduled to start as soon as freezing temperatures ceased. It was considered essential for these purposes to maintain the soil samples for all tests in a moist condition representative of the natural state of hydration in the early spring, but adjusting the moisture content to the conditions of each test.

The first step in this investigation was to obtain representative grain size analyses of these samples to disclose the character of the soils and the variations over the area and with depth. Each bag sample was placed in a large concrete pan. Weighings were made at all stages of the preparation of these samples for the various tests, so that complete evaluations could be made of the data. All of the rock fragments larger than the 1-in. sieve were removed from each sample, in preparation for other tests. The rock fragments were brushed clean of adhering moist soil with a wire brush and were weighed after air drying. A representative moisture content was taken from each pan after thoroughly mixing the material. The pans were then covered to prevent moisture loss until the soil was to be used in later tests. A sieve analysis was made on the entire rock portion removed from each sample below a maximum size of 4 in. permitted in the embankment down to the 1-in. size. The percentage of oversize material to be removed by the contractor from these borrow materials was determined for each sample and varied from a low value of about 2 per cent near the surface of the ground to about 12 per cent at a depth of 5 to 7 ft. A representative specimen of 3000 g of moist soil was selected from each pan after thorough mixing. The moist specimen was readily worked through the $\frac{3}{4}$-in., $\frac{3}{8}$-in. and No. 4 sieves by means of a stiff wire brush and a spoon to break up the lumps. The material retained on each sieve was thoroughly cleaned on the sieve by use of a wire brush. The moist soil passing the No. 4 sieve was weighed and a representative moisture determination was made on the thoroughly mixed material. The oven-dried fractions retained on the $\frac{3}{4}$-in., $\frac{3}{8}$-in., and No. 4 sieves were weighed. About 200 g of moist soil passing the No. 4 sieve was brought to a rather thin slurry condition in an evaporating dish by thoroughly mixing in water a little at a time thick enough to prevent the sand sizes from settling out. This procedure retained the natural state of hydration of the soil, as the representative condition. One-half of the slurry by weighing was transferred to the dispersion cup and the remainder was oven dried in order to determine the dry weight of the hydrometer specimen. The stirring rod method of dispersion was used for the final dispersion in the hydrometer jar, allowing the sand in the last 20 sec to settle out of suspension before the start of the hydrometer test. After completion of the hydrometer test, a sieve analysis was made on the sand fraction. Liquid and plastic limits were made on the moist soil passing the No. 8 sieve with its entire sand content, as being more representative of the natural plasticity characteristics and performances under field compaction conditions, particularly for determining whether the limitation of 25 per cent passing the No. 200 sieve was justifiable.

The grain size curves for depths of $1\frac{1}{2}$ to 4 ft and 5 to 7 ft are given in Fig. 2, together with the identifications of these soils, liquid and plastic limits, and plasticity indices, and other pertinent data. The grain size curves are essentially similar, but it is evident that at the greater depth of 5 to 7 ft there is a greater percentage of gravel sizes and of oversize material, indicating some decrease in weathering influences with depth. With the exception that the percentage of material passing the No. 200 sieve exceeded the 25 per cent limitation, these soils possess an almost ideal well-graded character for compactibility to high densities.

But actually these soils possess a very unusual character, which should have been fully evident in the handling and weighing of the sieve fractions, as essential information. The material passing the $1\frac{1}{2}$-in. sieve and retained on the No. 8 sieve was composed almost entirely of very light weight highly weathered but tough rock fragments, weighing only 1.2 g per cu cm (voids and solids) instead of about 2.8 g per cu cm of the solid rock (specific gravity). In order to understand the significance of this fact, the grain size curves of the $1\frac{1}{2}$ to 4 ft and 5 to 7 ft depths are recomputed on a volume basis using 1.2 g per cu cm for the material between the $1\frac{1}{2}$-in. and the No. 8 sieves, and value of 2.8 (specific gravity test) for all other materials. The changes in the percentage from a weight basis to a volume basis are indicated for the $\frac{3}{4}$-in., No. 8, and No. 200 sieve sizes. It is evident that the gravel content on a volume basis is very considerably larger than on the unrepresentative weight basis. Now the percentages passing the No. 200 sieve are less than the 25 per cent limitation. On this basis, at least, these materials would be acceptable as borrow.

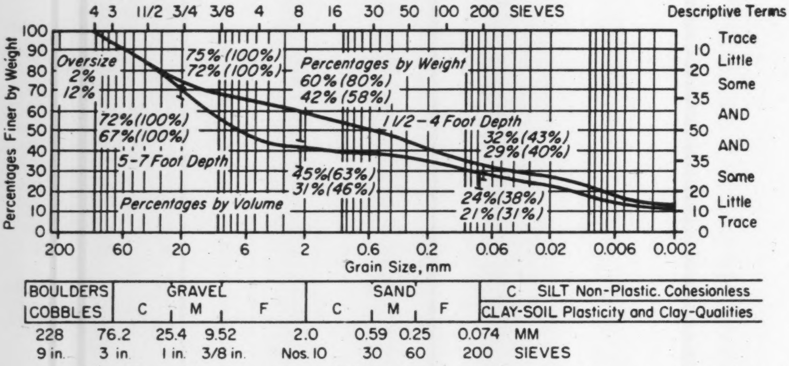


FIG. 2.—Grain Size Curves and Physical Characteristics of Borrow Materials.

Case 2.—Railroad Grade Crossing Elimination Embankments.

Identification ($1\frac{1}{2}$–2 ft) Orange-red coarse to fine SAND, some Silty Clay, and coarse to fine Gravel, trace cobbles. High PI.

(5–7 ft) Orange-red coarse to fine GRAVEL, and coarse to fine Sand, some Silty Clay, little cobbles. High PI.

Consistency limits of material passing No. 8 sieve
($1\frac{1}{2}$–2 ft) LL. 66.1 PL. 36.1 PI. 30.0. High plasticity.
(5–7 ft) LL. 72.5 PL. 38.1 PI. 34.4. High plasticity.
Original LL. 44.9 PL. 31.8 PI. 13.1. Medium plasticity.
Specific gravity of material passing No. 8 sieve 2.80
Unit dry weight of material between $1\frac{1}{2}$-in. and No. 8 sieves 1.20 g per cu cm

The low values of the liquid and plastic limits and plasticity index obtained from air-dried soil passing the No. 40 sieve in accordance with usual practices are compared with the considerably higher values obtained on the moist fully hydrated soil but containing all the sand to the No. 8 sieve, as representative of the natural condition. This indicates the marked effect of air drying on the state of hydration and probably also on the state of oxidation of the iron content of these soils. The moist soils appear to have more objectionable characteristics. However in the natural undisturbed state in the field in the zone of weathering influences, these soils possess an important natural high state of aggregation and structure, which requires considerable working and remolding to break down completely. As a consequence, together with the natural sand content, they exhibit natural plasticities in the relatively undisturbed state, as evidenced by their thread rolling capacity at the proper moisture content for this field test (3, pp. 432–433), of only low plasticity (P.I. 5 to 10). This in reality is a very favorable condition from the standpoint of construction operations of spreading the material in thin layers, and of compacting it to specified densities in the embankment, and of permanence of strength characteristics. If these materials are not overworked when too wet, their favorable characteristics would be retained during field compaction.

All this information was considered to be essential for interpreting properly compaction data for final acceptance or rejection of these soils on a rational and justifiable basis as suitable borrow materials and for purposes of formulating field criteria for control of compaction and for specification for construction of embankments.

Again it should be clear that no soil testing should be undertaken without adequate information and knowledge being supplied or obtained on the nature of the problems involved and on the conditions that control.

Case 3:

This case concerns the evaluation of the character of clay soils in a wide submerged bar across the mouth of a river, where consideration was being given to dredging a channel through the bar for shipping. Preliminary information was required from bottle sample of bar material obtained at two different depths along the line of the proposed dredged channel. The problem was to establish the character of these soils, (1) as influencing the dredging operations, where the dredged material by agitation dredging methods was to be discharged back into the water behind the dredge with the expectancy that currents would be strong enough to carry away the material before settling out; and (2) as influencing stable side slopes of the dredged channel, which would materially affect total quantities of dredging. These problems were fully discussed in conference. Eight samples from some 100 or more samples were selected at four typical locations across the bar at two depths each. These samples had been in the clients' office for some time and had been opened and handled, before the author was consulted.

The exposed surfaces of these samples were a brown color. By usual practices, specimens for hydrometer and consistency limit tests would have been taken from each one, thoroughly mixed, probably air dried, and used without much inspection or questioning. But the author's experiences with many different types of soils led him to question any brown color and to treat each new soil type, as a work of discovery, to learn its real character. Therefore wherever a core was available in a bottle, a portion was cut lengthwise with a fine wire saw and was examined. It was at once evident that the brown color was only skin deep (about $\frac{1}{4}$ in.) all over the samples, due to oxidation of ferrous iron typical of a marine environment toward the ferric state. The core of every sample was a striking light blue-gray color. The first step in the preparation of representative specimens was therefore to trim off with a fine wire saw all brown discolored material, and to use the moist material in order to make the testing fully representative of the natural marine environment. The grain size curves of two typical specimens are given in Fig. 3 by the (*a*) curves, which cover the approximate extreme spread of these soils.

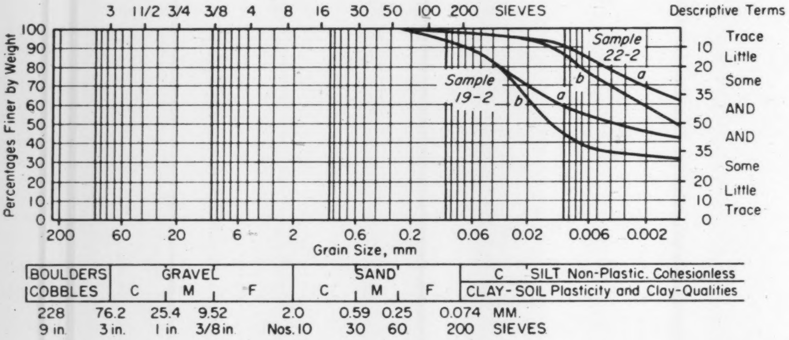


FIG. 3.—Comparison of Grain Size Curves and Consistency Limits of Representative and Altered Clay-Soil.

Case 3.—Submerged Bar Across Mouth of River.

Identification Sample 19-2 Light blue-gray Silty CLAY, trace fine Sand. High PI.
Sample 22-2 Light blue-gray CLAY, trace fine Sand. Very high PI.

Consistency limits and natural moisture content, as received

Sample 19-2	(*a*)	Original weight per cent 42.7	LL. 48.5	PL. 23.8	PI 24.7
	(*b*)	Altered	42.6	20.9	21.7
Sample 22-2	(*a*)	Original weight per cent 66.4	LL. 84.4	PL. 32.2	PI 52.2
	(*b*)	Altered	81.1	31.1	50.0

The trimmed off portions of the samples were replaced in the jars and sealed. Sometime later as a matter of interest to determine the influences of oxidation, these samples were re-tested. The results of these tests are given in Fig. 3 by the (*b*) grain size curves. It is evident that oxidation without actual drying out of specimens very materially affects and reduces the hydration characteristics of the soils, causing the material to appear to be more silty. This apparent difference in sedimentation characteristics would have been of importance and consequence with regard to agitation dredging methods. The material also appeared to be less plastic in the oxidized state.

Representative soil testing should require, as standard practice, the inspection of every sample after cutting a core lengthwise to reveal the portion of the specimen, which may or should be considered to be fully representative for testing. The unrepresentative portion should then be trimmed off. Otherwise the testing is certain to be unrepresentative and deficient in important respects. By following these representative procedures, as was done in six important projects and investigations, remarkable agreement was shown between predicted and observed performances of the soils.

Case 4:

This case concerns the testing and evaluation of the character and responses of varved silt and clay composed of alternating thin layers of variable thicknesses. Important problems were involved of settlement of five bridge structures and adjacent approach embankments due to consolidation, and of their stability conditions during and subsequent to construction. These problems (**5**, pp. 251–253) were fully discussed with the engineering consultants for planning subsurface explorations and for preparing adequate specifications for this exploration work.

Thirty-seven borings were made using a 2½-in. casing, two at each abutment and pier, from which bottle samples were taken at 5-ft interval of depth by a 2 by 1⅜-in. split barrel sampler. These borings were made under careful field supervision to insure high quality of the samples and information obtained. They served as exploratory borings to disclose the nature of subsurface soil conditions. On the basis of information obtained from at least two such borings at each bridge site, the location of the undisturbed sample borings was decided upon, where conditions appeared to be more critical. Six undisturbed sample borings were made, two at the main bridge site and one each at the other four sites. In the undisturbed sample borings 2-in. and 3-in. Shelby tube samples were taken alternately at 5-ft intervals of depth. All bottle samples were carefully identified (**3**, pp. 432–433) in the field by trained personnel of the engineering consultant. Soil profiles for each boring and bridge site were prepared and supplied for this investigation.

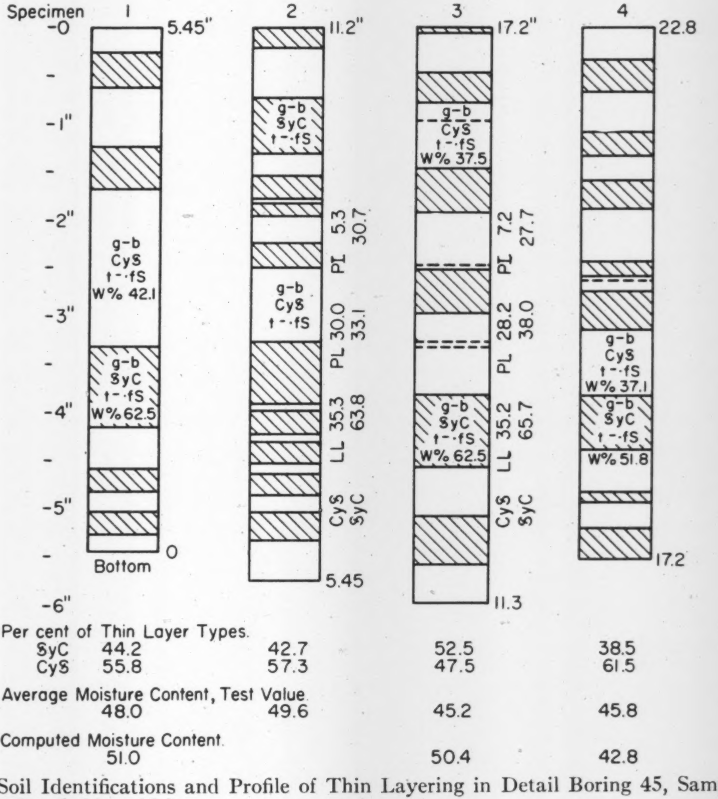
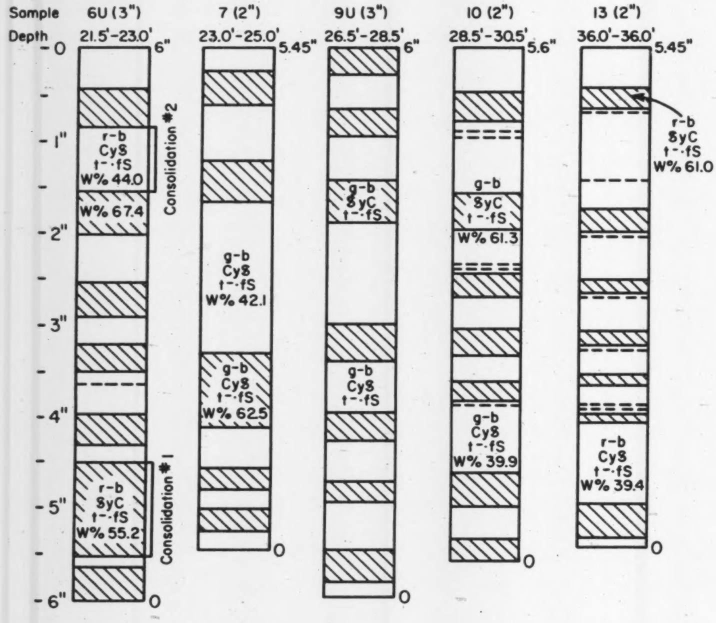

Specimen
1
2
3
4
-0
-1"
-2"
-3"
-4"
-5"
-6"
5.45"
11.2"
17.2"
22.8
0
Bottom
5.45
11.3
17.2
g-b
CyS
SyC
t-·fS
W% 42.1
W% 62.5
W% 37.5
W% 37.1
W% 51.8
CyS SyC
LL 35.3 63.8
PL 30.0 33.1
PI 5.3 30.7
LL 35.2 65.7
PL 28.2 38.0
PI 7.2 27.7
Per cent of Thin Layer Types.
SyC 44.2 42.7 52.5 38.5
CyS 55.8 57.3 47.5 61.5
Average Moisture Content, Test Value
48.0 49.6 45.2 45.8
Computed Moisture Content.
51.0 50.4 42.8

FIG. 4.—Soil Identifications and Profile of Thin Layering in Detail Boring 45, Sample 7, 2-in. Shelby Tube, Depth 23 to 25 ft.

All 2-in. Shelby tube samples from the undisturbed sample borings were identified accurately and in detail, in order to develop a representative, comprehensive and clear picture of subsurface soil conditions in the varved silt and clay stratum 2. Specimens about 6 in. in length were pushed out successively for the full length of the 2-in. tube samples. A slice was cut lengthwise from the side of each 6-in. specimen about ¼ in. thick. In first appearance the varves were practically indistinguishable, because the specimens exhibited a uniform brown color. After scribing inch marks along the length of the slice to allow for shrinkage adjustments and marking top and bottom positions, the slice was placed under a strong light with a reflector, which was positioned about 2 in. above the specimen. The remaining portion of the 6-in. specimen was placed in a bottle and covered to prevent moisture loss. The slice was dried (about 15 to 20 min) until the varves became clearly visible. The varves in this case were of two distinct layer types. The Clayey SILT varves dried out to a light color, while the Silty CLAY varves remained a dark moist color and stood out in bold relief with sharply defined interfaces. The profile of each 6-in. specimen was accurately recorded in sequence in proper top and bottom position, showing in detail the varve character, thicknesses, and spacings by placing the record sheet alongside of the partially dried slice, and by drawing the varve interfaces. Compensation was made for shrinkage through the 1-in. marks, the major part


Sample
Depth
6U (3")
21.5'-23.0'
6"
7 (2")
23.0'-25.0'
5.45"
9U (3")
26.5'-28.5'
6"
10 (2")
28.5'-30.5'
5.6"
13 (2")
36.0'-36.0'
5.45"
- 0
- 1"
- 2"
- 3"
- 4"
- 5"
- 6"
r-b CyS t-·fS W% 44.0
W% 67.4
Consolidation #2
r-b SyC t-·fS W% 55.2
Consolidation #1
g-b CyS t-·fS W% 42.1
g-b CyS t-·fS W% 62.5
g-b SyC t-·fS
g-b CyS t-·fS
g-b SyC t-·fS W% 61.3
g-b CyS t-·fS W% 39.9
r-b SyC t-·fS W% 61.0
r-b CyS t-·fS W% 39.4
0

	6U (3")	7 (2")	9U (3")	10 (2")	13 (2")
Per cent of Thin Layer Types					
SyC	53.2	44.2	39.0	44.5	33.3
CyS	46.8	55.8	61.0	55.5	66.7
Average Moisture Content, Test Values					
	54.0	48.0		45.6	45.2
Computed Moisture Content					
	56.4	51.0		49.5	46.6

FIG. 5.—Soil Identifications and Profile of Varved Deposit With Thin Layering in Detail for Bottom Specimens of Samples of Boring 45.

of the shrinkage occurring in the Silty CLAY varves.

The remaining undried portion of each 6-in. specimen was then placed alongside of the partially-dried slice in proper top and bottom position and was accurately scribed at each varve interface. The varve segments were then cut by means of a fine wire saw. The varves were separated into the two layer types, and each varve was scraped clean by a spatula of the adjacent dissimilar layer type, which was readily distinguishable in the scraping process. The two layer types were placed in separate widemouth half-quart jars in proper sequence for careful identification of each layer type by visual and manual methods, and moisture contents for each 6 in. specimen. A few hydrometer tests, and liquid and plastic limit tests were made on moist soil without air-drying for accurate verification of character and identification of layer type.

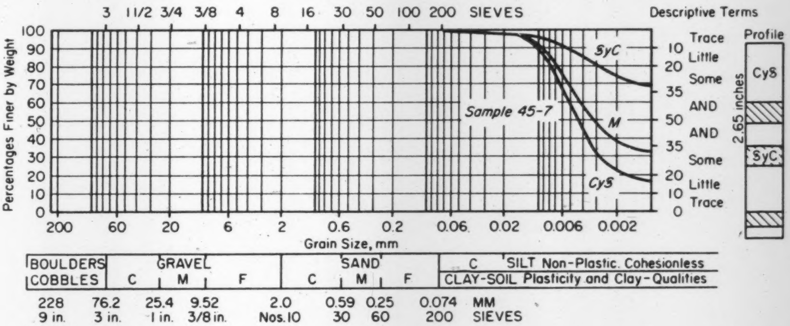


FIG. 6.—Representative Testing for Grain Size Curves and Consistency Limits. *Case 4.*—Detailed Investigation of Character of Varved Silt and Clay.

Identification Varved Brown Silty CLAY, trace fine Sand. High PI. 30.5 per cent
and Brown Clayey SILT, trace fine Sand. Low PI. 69.5 per cent
Consistency limits
SyC Varve LL. 63.8 PL. 33.0 PI. 30.7 Percentage of layer type 30.5 per cent
CyS Varve LL. 35.3 PL. 30.0 PI. 5.3. Percentage of layer type 69.5 per cent
Mixture M computed LL. 44.0 PL. 31.0 PI. 13.0

Computed grain size data for mixture at		0.006 mm		0.004 mm		0.002 mm
SyC Varve 30.5 per cent	93 per cent	28.4	84 per cent	25.6	72 per cent	22.0
CyS Varve 69.5 per cent	70 per cent	48.7	45 per cent	31.2	21 per cent	14.6
Mixture M percentages		77.1 per cent.		56.8 per cent		36.6 per cent

A typical soil profile from four 6-in. specimens from a single Shelby tube sample is given in Fig. 4. It shows in detail the varved character and identifications of layer types, layer thicknesses and spacings, moisture contents, and liquid and plastic limits of representative layer types. At the bottom of the figure are given the proportions of each layer type. It is evident that while there are variations in detail in layer thicknesses and spacings, the proportions of each layer type in each specimen are comparable. A typical soil profile for the entire thickness of the varved stratum 2 for this same boring is given in Fig. 5, using the bottom specimen only for each sample. There is now evident a significant variation in layer type, and in proportions with depth in stratum 2. Such information is essential in disclosing adequately the representative character of varved or thin-layered soils for investigations of this character.

By ordinary common practices, the varved character might have passed unnoticed, being almost indistinguishable in the undisturbed moist specimens, or if it had been recognized, average specimens would have been considered sufficiently useful in accordance with common conceptions and practices for all practical purposes for moisture contents, hydrometer tests, and liquid and plastic limit tests. For comparative purposes to bring out clearly in perspective the marked differences in significance and adequacy of these two approaches, average moisture contents were obtained from a number of specimens by cutting a portion lengthwise from a 6-in. specimen. Typical values are given at the bottom of Fig. 4. Computed average moisture contents by direct proportion methods are also given, as noted, which were based on the moisture contents and proportions of the two layer types. It is evident that the computed values check sufficiently closely the test values for practical purposes. This means that a reasonable estimate can always be made of the average moisture content, if required for some purpose. But, because of the characteristic and marked differences in the moisture contents of the two layer types, it should be clearly evident that it would be impossible to deduce these moisture contents from the average moisture content alone.

In Fig. 6 are given for comparative purposes the grain size curves obtained from hydrometer tests of the two separated layer types- Clayey SILT and Silty CLAY, and of a composite average specimen indicated in the figure. The composite curve can be estimated almost exactly by taking proportions of the curves of the two layer types at a number of particle sizes to define the grain size curve for the composite material. Again, it would be impossible to deduce the grain size curves of the individual layer types from the curve of an average specimen of indeterminate unknown proportions. Similarly, liquid and plastic limits, and plasticity indices, obtained from each layer type, are given in Fig. 6. All this representative information is essential for clearly visualizing and evaluating the conditions that control and for appraising significantly their influences on the over-all responses and performances of these soils in the deposit in this investigation. By proper programming and scheduling of the testing in sequence, there need not be any delay or time loss in carrying out the program.

It should become standard practice in testing of varved and thin-layered soils to follow such procedures and to provide complete and fully adequate and representative test data and information, such as shown in Figs. 4, 5, and 6. The use of averages in such investigations always penalize the favorable aspects inherent in a situation and obscure the unfavorable and adverse aspects, so that they cannot be properly recognized and adequately taken into account in investigations for engineering works. The major emphasis in conceptions and practices should be placed on being representative and adequate.

PROPERTY AND STRENGTH TESTS

An appraisal and evaluation of the environmental and imposed conditions was made with regard to their nature and their influences on soil responses in compaction and consolidation tests. These were translated into significant test conditions, which would reproduce representatively their controlling influences.

Case 2:

The preliminary investigations for this case were considered earlier. Compaction tests were made in order to serve as a reliable and adequate basis for final acceptance or rejection of this borrow material, and, if acceptable, for establishing criteria for compaction control in the field and for preparing specifications for construction.

Because of the high degree of compaction needed for a stable fill for a railroad with a minimum of settlements subsequent to construction, the modified A.A.S.H.O. compactive effort was used as being representative of the required field compaction. Compaction tests were performed on two large composite samples of about 75 lb each of material passing the 1-in. sieve, which were taken from the more centrally located bulldozer cut at depths of 1½ to 4 ft and 5 to 7 ft. Due to the large gravel size content, especially by volume percentages, the use of a 6-in. mold was considered essential and more representative. This soil had not been permitted to dry out as a basic condition. The common practice of substituting an equal percentage of material passing the 1-in. sieve and retained on the No. 4 sieve for the coarser sized material retained on the 1-in. sieve to 4-in. maximum size, as a very rough approximation of the whole soil, was considered to be entirely unrepresentative and unpermissible: (1) it would markedly change the gradation characteristics of the soil and hence the compaction characteristics and maximum densities in an unknown and uninterpretable way, and (2) it therefore would also change the wetting characteristics of the material retained on the No. 4 sieve and hence moisture requirements and optimum moisture for maximum density, which would bear little relation to the permissible range of moisture of the real soils.

In order to preserve the proper moisture condition of the porous light weight material between the 1-in. and No. 8 sieve, as representative of the natural drying out of the deposit with advance in the season from spring to summer conditions and of normal moisture losses by evaporation during handling and processing the soil while compacting it in the embankment, the compaction test was started at approximately the field moisture content then prevailing, yielding the first moisture-density point on the curve. The entire batch of soil in each sample was then partially dried for each successive test point, by frequent mixing and overturning of the sample with a trowel, each test defining a point on the moisture-density curve. The moisture loss was checked at intervals by weighings on a small pan of soil buried to a depth level with the surface of the sample in the large pan. The slowness of the drying, especially in the first stages, was indicative of conditions, which might be expected in the field, where over-wet material had been brought on to the embankment and had to be processed to bring the moisture content within permissible limits. This is considered essential information for field control.

The re-use of compacted material was considered unrepresentative. Therefore the oven-dried specimens for moisture content determinations were discarded. In order to obtain representative moisture determinations, especially in this case with the large percentage of material retained on the No. 8 sieve of a lightweight and porous character, the entire 6-in. mold sample was used after performing a California Bearing Ratio (CBR) test. At night the pans were covered to prevent additional moisture losses and to permit attaining a uniform distribution of moisture throughout the sample at this stage of the testing. This procedure did not materially lengthen the total time of all testing because the various operations and tests discussed previously were scheduled to dovetail sufficiently, so that while waiting for the batches to dry progressively between compaction tests to the desired moisture point, other tests were being performed. CBR tests were performed on both top and bottom faces of each specimen to determine the upper limit of moisture content permissible for compaction in the embankment, which would permit attaining the specified compaction, the movement of hauling and spreading equipment, and the conditioning of the soil to proper moisture content, if wetter than specified.

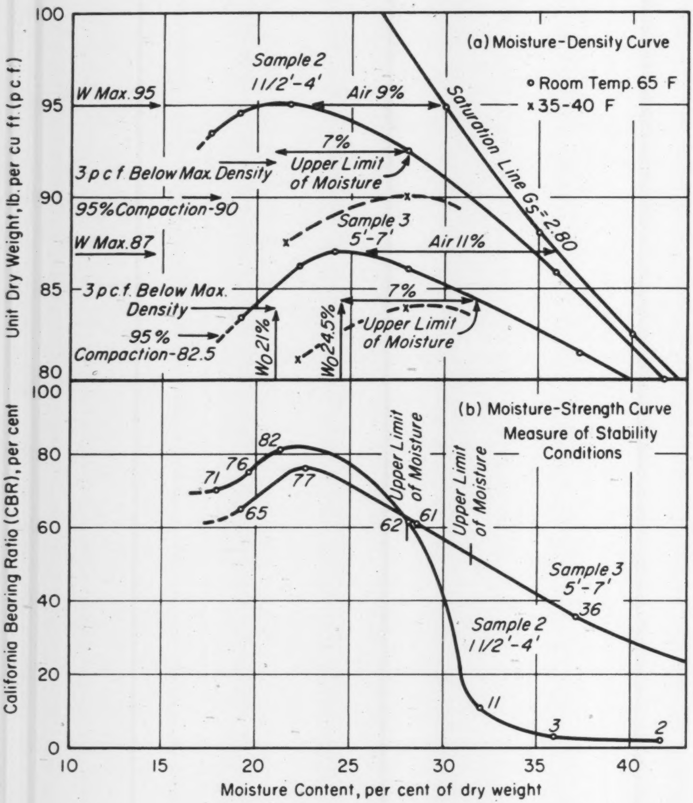


FIG. 7.—Moisture-Density-Strength Relations for Soils of the Century Borrow Pit Area. Upper Area, Center Bulldozer Cut.

The moisture-density curves for room-temperature conditions, about 65 F, are given in Fig. 7 for these two specimens. In order to properly take into account and to provide representative and reliable criteria for field control of compaction for the early spring work, when freezing temperatures ceased, a few duplicate compaction tests were made at representative temperatures of 35 to 40 F. This was accomplished by placing the covered pans of soil out-of-doors at night. The resulting compaction curves are shown by dotted lines with about 5 lb per cu ft decrease in maximum density for the sample at depths of $1\frac{1}{2}$ to 4 ft with less gravel, and about 3 lb decrease for the sample at depths of 5 to 7 ft with the greater gravel content. The peak density in both cases was at a higher moisture content. Where soils are to be compacted in the early spring or late fall, such additional representative data is essential for establishing reliable and adequate criteria for field control and should be required as standard practice.

The compaction properties of these two soils, due to their unusual characteristics, required careful interpretations as a basis for properly judging their suitability as embankment materials.

1. Due to the light weight of the weathered rock, the sample from the 5 to 7 ft depth containing the higher percentage of material retained between the $\frac{3}{4}$ in. and No. 4 sieves (42 per cent by weight and 54 per cent by volume in Fig. 2) yielded a lower maximum density of 87 lb per cu ft, whereas the sample from the $1\frac{1}{2}$ to 4 ft depth containing the lower percentage (20 per cent by weight and 37 per cent by volume) yielded a higher maximum density of 95 lb per cu ft. In order to compare these maximum densities with those of normal materials, the maximum densities were adjusted for a specific gravity of the material between the $\frac{3}{4}$-in. and No. 8 sieves equal to that of the remainder of the soil (1.2 g per cu cm to 2.7 g per cu cm). For the $1\frac{1}{2}$ to 4-ft depth sample the 95 lb per cu ft became 133, and for the 5 to 7 ft depth sample the 87 lb per cu ft became 144, which would be considered to be very good to excellent densities.

2. The amount of air-voids percentages in the soils is large but is really the sum of the normal air voids trapped in a compacted clay soil plus air voids contained in the porous lightweight rock material, as it partially dried out in reaching the maximum density condition.

3. The stability conditions of the compacted soils on the wet side of the peak or maximum density, as indicated by the CBR values for the as-compacted condition, do not drop adversely, due to their favorable natural state of aggregation and structure, until the moisture content exceeds the optimum moisture content at maximum density by more than 7 per cent. Part of the moisture retained in the porous rock material is evidently not available in decreasing adversely the consistency and strength of the compacted soil for operations of processing and compacting the material in the embankment. For these purposes it was considered on the basis of rather common experiences that the CBR value should not drop below a value of 10 when compacting on the wet side of the peak of density. Therefore the 7 per cent moisture limit in excess of the optimum moisture was set as the maximum for material being brought onto the embankment. The relatively high values of CBR for moisture contents smaller than this limit are indicative of the relatively stable characteristics of these compacted soils.

4. Although the rock material between the 1-in. and the No. 8 sieves is a lightweight porous material, it did not appear to be broken up and unduly crushed by the heavy compactive effort of the modified A.A.S.H.O. compaction in the test, and therefore it may be expected to perform similarly in the field compaction.

5. In the new environment imposed in the compacted embankment with normal ground water below the surface of the original ground on which the embankment is to be constructed, and with normal seasonal saturation by precipitation considerably reduced, especially with the contemplated effective drainage of the ballast fill under the railroad tracks, the normal weathering processes would be almost eliminated or

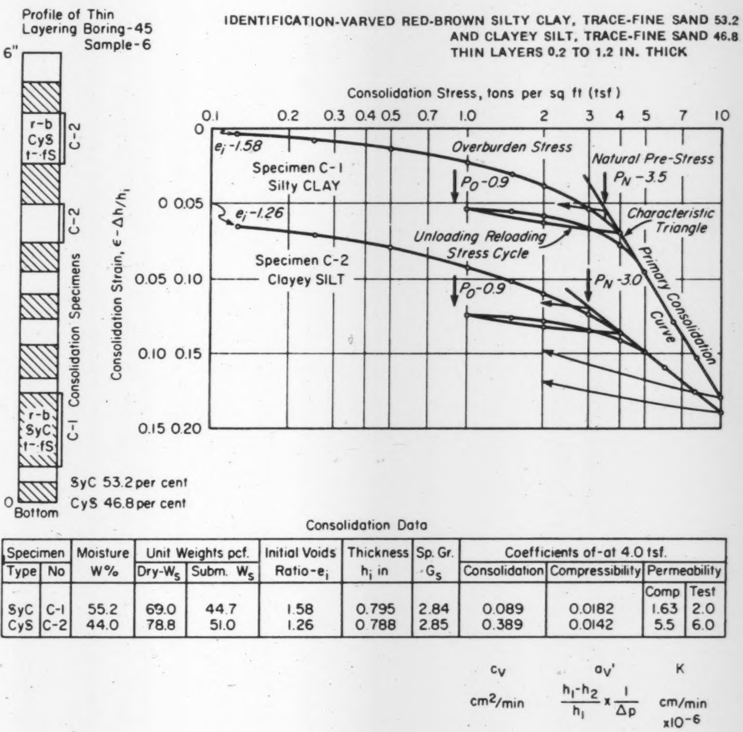
be greatly reduced. These weathered rock fragments composing the large part of the material should therefore be permanently stable under this new environment.

It is evident that these relatively favorable aspects discovered in the test and made interpretable by representative testing could be properly weighed and evaluated as essential information in comparison with the apparently more obvious rather unfavorable aspects in finally judging the suitability and acceptability of these soils for the embankment construction. The spring of the year was obviously the more critical condition with regard to the relatively high moisture content of the material. Conditions could be materially improved by running deep furrows by means of a rooter plow up and down the hillside slope at regular intervals across the slope in order to permit the excess free water in the soil to drain out through its favorable natural state of aggregation and fissured structure. These principles of representative testing of soils have been applied in the soil investigations and in the recommendations for field control of compaction and construction and for design of three flood control earth dams and of three water supply earth dams.

Case 4:

The preliminary investigations for this case were considered earlier, and the results were illustrated in Figs. 4, 5, and 6, which have disclosed representatively the character of the varved deposit. The principal objectives of this investigation were to determine adequate and representative information on consolidation and shearing strength properties of these varved soils for the purpose of evaluating conditions that control, and for making settlement and stability analyses.

This required that representative consolidation stress-strain properties and time-consolidation properties be determined by a consolidation test on each layer type, that is, two tests for each 3-in. Shelby tube sample tested. This was accomplished by using the procedures followed in identifying the varve types. When the varves became distinctly visible, specimens of individual layer types were marked out on the undisturbed portion of the 6-in. specimen by matching against the now visible varves in the dried slice. If specimens of each layer could be found thick enough for a consolidation specimen, that is, about 0.6 to 1.0 in. thick, they were cut out by a wire saw and were carefully trimmed and fitted into a 2.5-in. consolidation ring of the floating ring type. At first, if all layers were too thin, a combination of two layer types was chosen to bracket conditions—(*a*) a combination with a Clayey SILT varve on each outside and with a thin Silty CLAY varve in the middle, and (*b*) a combination with a Silty CLAY varve on each outside and with a thin Clayey SILT varve in the middle. It was found by analyses of two sets of data obtained from such composite specimens that this procedure was not sufficiently representative of the stress-strain and time-consolidation properties of the layer types. It was therefore abandoned as unsatisfactory after two sets of such tests. In most cases by pushing out a second 6-in. specimen, a thick enough layer could be found of each layer type. If one of the 6-in. specimens was not used for a layer type for consolidation tests, the specimen was tested in triaxial compression to determine the shearing strength properties of the varved soil. The triaxial test on a representative composite specimen of varves would be representative of shearing conditions, where the slip plane actually crossed the varves at an angle (**5**, **8**). In a few cases where no layer could be found thick enough for a consolidation specimen, a specimen was obtained by planing off one face each of two varves of the same type carefully in a consolidation ring with a metal screed and joining these two faces to form a sufficiently thick specimen. Comparative tests showed that when the planing and joining was carefully done, representative and comparable results could be obtained.



Consolidation Data

Specimen		Moisture	Unit Weights pcf.		Initial Voids	Thickness	Sp. Gr.	Coefficients of-at 4.0 tsf.			
Type	No	W%	Dry-W_s	Subm. W_s	Ratio-e_i	h_i in	G_s	Consolidation	Compressibility	Permeability	
										Comp	Test
SyC	C-1	55.2	69.0	44.7	1.58	0.795	2.84	0.089	0.0182	1.63	2.0
CyS	C-2	44.0	78.8	51.0	1.26	0.788	2.85	0.389	0.0142	5.5	6.0

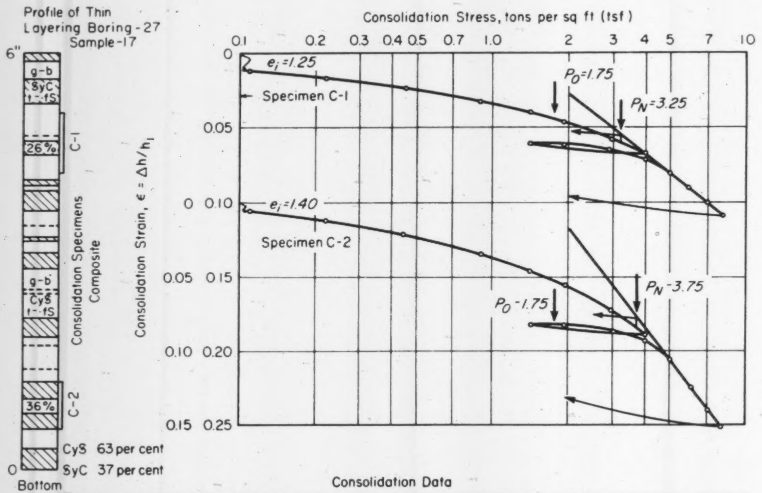
c_v a_v' K

cm²/min $\frac{h_1-h_2}{h_1}\times\frac{1}{\Delta p}$ cm/min $\times 10^{-6}$

FIG. 8.—Consolidation Stress-Strain Curve. Boring 45, Sample 6U, Depth 23.0 ft.

Typical consolidation stress-strain curves are given in Fig. 8 for specimens of layer types obtained from the same boring as that tested in Figs. 4, 5, and 6. The soil profile of the 6-in. specimen used for the consolidation specimens is given in Fig. 8 with the locations of the specimens noted. Pertinent data obtained from the consolidation test are also given in the figure, so that complete information is available for purposes of interpretation and use. Typical consolidation stress-strain curves are given in Fig. 9 for composite specimens, as discussed above, with the specimens shown on the soil profile bracketing varve conditions. However, it is evident by comparison with Fig. 8 that these curves are unrepresentative and uninterpretable.

In the consolidation testing, procedures were selected with certain modifications (**6**, pp. 212–220) (**7**, pp. 83–89), which were considered to be essential and representative of the conditions disclosed by this investigation. The first consolidation test had shown that the varved soil had been over-consolidated to a maximum pre-stress of 3.5 to 4.5 tons per sq ft at some time in its geological and stress history. To eliminate the possibilities of any disturbance effects to the soil structure due to swelling of the Silty CLAY varves in contact with water, the submergence of these specimens was deferred until the overburden stress was restored on the specimen in steps, but with the specimen protected against moisture losses by evaporation. Each stress increment was allowed to remain in place for at least 24 hr.



Consolidation Data

Specimen No	Moisture W%	Unit Weights pcf.		Initial Voids Ratio-e_i	Thickness h_i in	Sp Gr G_s	Coefficients of-at 4.0 tsf			
		Dry W_s	Subm-W_s'				Consolidation	Compressibility	Permeability	
									Comp.	Test
C-1	42.8	79.0	51.1	1.25	0.796	2.84	0.67	0.0104	6.7	6.9
C-2	49.3	74.0	48.0	1.40	0.547	2.84	0.23	0.156	3.6	4.7

c_v cm^2/min a_v' K-cm/min $\times 10^{-6}$

FIG. 9.—Consolidation Stress-Strain Curves. Boring 27, Sample 17U, Depth 47.5 ft.
IDENTIFICATION-VARVED GRAY-BROWN CLAYEY SILT, TRACE-FINE SAND 63 PER CENT AND SILTY CLAY, TRACE-FINE SAND 37 PER CENT

The loading conditions and sequences varied over the project, certain bridges requiring additional approach fill adjacent to the abutments, other requiring excavation to grade elevations, and others requiring little change from present ground elevations. To take into account properly these different loading conditions and sequences and thus to obtain consolidation data having direct and representative applications to these specific cases, the specimens were first of all loaded in increments of stress to just beyond the natural pre-stress, p_N, and then were unloaded in stress increments to the present overburden stress p_o at the sample elevation. The stress sequence thereafter was made to follow representatively the probable loading conditions. Where additional approach fill was to be placed or where little change in conditions was contemplated, the specimens were reloaded in increments to 10 tons per sq ft. Where additional excavation was required to grade elevations, the specimen was unloaded further to a stress representative of conditions in the deposit at the elevation of the particular sample resulting from the unloading of the deposit by excavation at the bridge site.

The stress increments beyond a stress of one ton per sq ft were made smaller, as noted in Fig. 8, than the general practice of doubling the stress. The double stress increments are considered to be unrepresentative of the probable actual loading conditions because of disturbance effects to the soil structure under the larger load increments. The unloading-reloading stress cycle is considered to be essential: (1) for definitely, representatively, and adequately establishing these stress-strain relations for direct use in settlement analyses, where the soils are over-consolidated or where excavations are to be made, which produce or increase a state of over-consolidation, and (2) to establish representatively and reliably the time-consolidation relations for direct use in estimating the time-delay of consolidation to be expected. Furthermore the unloading-reloading stress cycle defines a characteristic triangle at its incidence to the linear primary consolidation curve, which permits establishing the natural pre-stress, p_N reliably.

An analysis of the time-consolidation relations for the Clayey SILT layer type showed that neither the Taylor method of square root plotting of the time data, nor the semi-log plotting yielded permeability data, which was representative; therefore direct permeability tests were made on all consolidation specimens at each load increment on the reloading stress cycle and thereafter to establish conditions properly. These permeability tests showed that the permeabilities of the Clayey SILT layer type were considerably greater than estimated from the coefficient of consolidation.

The undisturbed character of these varved soils is indicated in Figs. 8 and 9 by the small strain at $\frac{1}{8}$ ton per sq ft and by the relatively flat laboratory reloading curve to the natural pre-stress. The high value of the pre-stress of 3.5 to 4.5 tons per sq ft is a very favorable aspect in this situation, materially reducing consolidation settlements and increasing stability conditions. The amount of unloading of the deposit to the present overburden stress, as indicated in Fig. 8, means that the very flat reloading stress cycle from the present overburden stress up to about the point where it crosses the unloading stress cycle controls the settlements relations, a very favorable aspect indeed. The real nature of the compressibility of the Silty CLAY and Clayey SILT varves is revealed by the respective consolidation stress-strain curves, the Clayey SILT layer type being about one-half as compressible, especially on the reloading stress cycle. The settlements contributed by the varved portion of the deposit can be estimated with considerable real accuracy and reliability on the basis of such typical consolidation curves and on the basis of complete determinations of the percentages of each layer type from soil profiles, such as shown in Figs. 4 and 5. In addition, due to the influences of the stressing on the reloading stress cycle, as representative of actual conditions, and the consequent decrease in the coefficient of compressibility and corresponding increase in the coefficient of consolidation with somewhat minor decreases only in permeability, the time-delay of consolidation will be considerably shorter, especially due to lateral consolidation drainage along the very much more permeable Clayey SILT layers. This is an additional very favorable aspect.

These principles of representative consolidation testing of varved or thin-layered clay soils have been applied in the investigations of many important problems during the past 20 years, where such favorable aspects were disclosed for purposes of design and construction of structures, particularly for Drydocks 5 and 6 in the New York Navy Yard during 1941 to 1943, where some 160 samples of varved silt and clay were identified in detail and representatively tested for their strength properties; for The White House reconstruction in 1952, which was underlaid by a thin-layered clay and silt; for the Hudson River crossing of the New York State Thruway from Tarrytown to Nyack, where a thick layer of varved silt and clay was encountered at considerable depths in the river deposit; and for two cases, where thin fine sand layers and partings materially affected and influenced favorably consolidation drainage conditions in the horizontal direction in the organic silt of the Hudson River at Pier No. 57 of the buoyant box construction, and at the pier sites of the Kingston-Rhinecliff Bridge.

CONCLUSIONS

Representative soil testing can disclose significantly the favorable and unfavorable aspects inherent in a situation so that they can be recognized and properly evaluated. Full advantage can then be taken of the favorable aspects by fitting design and construction methods more closely to actual existing conditions. The possibilities can be explored for improving any unfavorable conditions, but in any case they can be fully taken into account by establishing in advance safe limits and time sequences for design and construction. But in order to accomplish these important objectives, it is essential that the nature and the complexities of the soil problems involved in an investigation and of the conditions that control soil behavior be fully recognized and appraised, both by those requesting soil tests to be made and by those performing the soil tests (3, pp. 408) (5, pp. 251–253).

It should be fully realized that soils are very different from the common structural materials where simple acceptance tests are the rule, because soils, from the nature of their character and responses, are strongly susceptible to the influences of environmental conditions and to changes in conditions and treatment to which the soils may be subjected in the construction of structures Full exchange of essential information, opinions, and test findings should be made available during the testing program. So often the real nature of the conditions that control and their influences are revealed only during the progress of the soil testing. Those making the soil tests have a responsibility to recognize significant conditions and responses and to supply essential information on these conditions to those requesting the soil tests, so that they can be studied and evaluated before the testing program has been completed, thus permitting more complete evaluations. But competent soil mechanics advice must be obtained, judgment must be exercised, and a proper share of responsibility must be accepted by both parties, in order to obtain fully adequate, representative, and reliable soil test data, which has valid and direct applications in the investigation under consideration. Adequacy here is an essential prerequisite to adequacy in the final outcome of an investigation, and hence in the final decisions of design and construction. This requires soil testing of a higher order.

The major emphasis in soil investigations for engineering works should be on attitudes of "works of discovery" and on higher concepts and practices of adequacy in soil investigations. Thus it would be possible to realize high standards of excellence and significant economies in the planning, design, and construction of foundations of structures and of earthworks.

The most important forward step for the future in realizing these practical engineering objectives is: *To treat and to test real soils under influences of essentially real and representative conditions that control.*

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EVALUATING RELATIONSHIPS BETWEEN MOISTURE CONTENT, DENSITY, AND VOID PROPERTIES OF SOILS*

By Norman W. McLeod[1]

Synopsis

While soil composition is usually expressed on a weight basis, a better understanding of a number of soil properties is obtained by analyzing it on the basis of volume. For soils consisting of particles that absorb little or no water, this volumetric analysis can be made simply and accurately. However, for relatively absorptive, dense-graded, well-compacted, coarse-grained soils, correct values for void ratio, air voids, and degree of saturation can be obtained only when the volumetric analysis is based on the ASTM bulk specific gravity of the soil particles and when full allowance is made for the water absorbed into the water-permeable pore space within the individual particles of soil. The paper includes several simple charts that contain families of curves relating the five variables, moisture content, density, void ratio, degree of saturation, and per cent air voids, for any homogeneous soil mass. Density and stability curves can be plotted on these charts, which could be useful in phases of soil engineering pertaining to soil compaction, consolidation, permeability, capillarity, sand or gravel filters, drainage facilities, frost action, etc. These charts have the time-saving advantage that after the point representing the moisture content and density of a soil sample has been plotted on the appropriate chart, correct values for the void ratio, degree of saturation, and per cent air voids of the sample can be read directly from the chart by interpolation.

Soil masses consist basically of soil and water, or of soil, water, and air or other gas. It is common practice to express the composition of soil on a weight basis. The weight of the soil itself is usually reported as pounds of either oven-dry or wet soil per cubic foot of bulk volume of the soil mass. Soil moisture content, on the other hand, is ordinarily expressed as a percentage of the oven-dry weight of the soil (grams of water per 100 g of oven-dry soil).

While expressing soil composition on a weight basis has certain practical advantages, this approach can be quite misleading in several respects when soils vary widely in specific gravity, and it fails to provide certain basic information. For example, mere weight values for the soil and water components provide little indication of the degree of saturation of a soil, its void ratio, or per cent air voids. Nor do they indicate how these void properties would change with wide variations in either the specific gravity of the soil, or its moisture content, or the bulk density of the soil mass. Consequently, for a better understanding of the proper-

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ties of soils, and of their performance in engineering structures, it is worth while to analyze soils on a volumetric basis.

Volumetric Basis for Analyzing Soils

The volumetric approach to soil analysis is illustrated by Figs. 1, 2, and 3. Saturated soils consist of only soil and water. In this case, all the water-permeable porosity within the individual soil particles, together with the entire intergranular space between the particles, is filled with water. Figures 1, 2, and 3 illustrate the soil, water, and air or other gas content of unsaturated soils. Figures 2 and 3 pertain to unsaturated soils consisting of particles without and with internal water-permeable porosity, respectively.

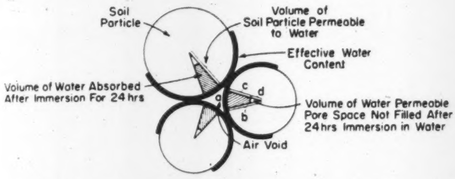
Soil Particle
Volume of Soil Particle Permeable to Water
Effective Water Content
Volume of Water Absorbed After Immersion For 24 hrs
c
d
a
b
Volume of Water Permeable Pore Space Not Filled After 24hrs Immersion in Water
Air Void

Fig. 1.—Illustrating Factors to be Considered When Analyzing Soils on a Volumetric Basis.

The relative proportions by volume of soil and water, or of soil, water, and air, can vary over very wide limits. In a dense-graded, well-consolidated, gravel base course on a highway or airport, the soil material can occupy as much as 85 to 90 per cent of the bulk volume of the base course. On the other hand, in certain cellular and other very open soil structures, the volume occupied by the soil can be much less than 50 per cent of the bulk volume of the soil mass.

Three Specific Gravities Employed for Soils

At the present time, two specific gravities are commonly determined for coarser soil particles such as gravel and sand: ASTM bulk specific gravity and ASTM apparent specific gravity.[2]

According to the definition for ASTM bulk specific gravity, the volume of a soil particle includes the volume of the water permeable pore space within the particle and is represented by any one of the three full circles in Fig. 1. On the other hand, ASTM apparent specific gravity separates the bulk volume of a coarse soil particle (full circle in Fig. 1) into two components. One of these is the volume of internal pore space into which water can penetrate during immersion for 24 hr, represented by the partial wedge *c* in Fig. 1. The other component is the volume of material that is impermeable to water during 24 hr of immersion, the full circle minus the partial wedge *c* in Fig. 1. When the individual particles contain no water-permeable pore space, Fig. 2, their ASTM bulk and ASTM apparent specific gravities become equal.

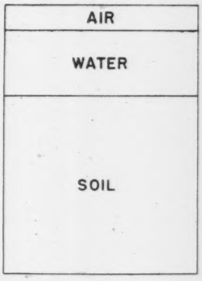

Fig. 2.—Soil Composition on a Volumetric Basis (No Absorption of Water into the Soil Particles).

The test for ASTM apparent specific gravity limits the water absorption period to 24 hr. A longer immersion time might indicate the existence of additional water permeable porosity, illustrated by *d* in Fig. 1.

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[2] Standard Method of Test for Specific Gravity and Absorption of Coarse Aggregate (C 127), and Fine Aggregate (C 128), 1955 Book of ASTM Standards, Part 3, pp. 1231 and 1233.

When soil particles are quite small, the amount of water-permeable pore space within the individual particles cannot be determined accurately by present methods. However, it is believed that the current procedure for measuring the specific gravity of fine-textured soils, ASTM Method D 854,[3] which involves soaking in water for 12 hr, provides values closer to ASTM apparent than to ASTM bulk specific gravity. Only when the particles of a fine textured soil are impermeable to water does its measured specific gravity by ASTM Method D 854 become also its ASTM bulk specific gravity.

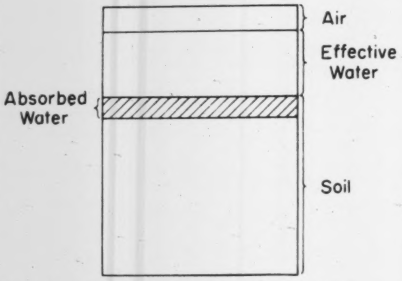

FIG. 3.—Soil Composition on a Volumetric Basis (Some Absorption of Water into the Soil Particles).

Water Absorption by Soil Particles

The absorption of water into the water-permeable pore space within individual soil particles does not always seem to receive the attention that it merits. This is understandable insofar as fine-textured soils are concerned, since there is no standard method available at present for measuring the internal water-permeable porosity of the individual grains. However, this information can be obtained for the coarser-grained soils like the gravels and sands that are commonly used for base courses for highways and airports, drainage installations, filters, etc. A recent paper by Ricketts, Sprague, Tabb, and McRae (**1**)[4] lists water absorption values ranging from 0.2 to 11.7 per cent for about three dozen different aggregates. It is apparent, therefore, that the amount of water that must be added merely to fill the water-permeable pore space within the individual particles of water-absorptive gravels and sands can be considerable. Consequently, measurement of this water absorption can lead to a better understanding of the design, construction requirements, and performance of earthwork structures for which these coarser-grained soils are employed.

Void Properties Employed in Soil Engineering

The technical literature on soil engineering refers to three different void properties of a soil mass: "void ratio," "per cent air voids," and "degree of saturation." "Void ratio" is defined as the ratio of the volume of intergranular space to the volume of solid particles in a given soil mass.[5] "Per cent air voids" is defined as the total volume of the intergranular air spaces, a in Fig. 1, expressed as a percentage of the bulk volume of a given soil mass (**2**). "Degree of saturation" is defined as the volume of water expressed as a percentage of the volume of the intergranular space in a given soil mass.[5]

The values calculated for these three void properties depend upon which measured specific gravity is selected for the soil, that is, ASTM bulk, ASTM apparent, or ASTM D 854 specific gravity, and upon the quantity of water absorbed into the water-permeable pore space (*b* in Fig. 1 and the cross-hatched area of Fig. 3) within the individual soil particles. The "void ratio" is larger when based on ASTM apparent than on ASTM bulk specific gravity. A portion or all of the water-permeable porosity of the individual soil grains may be included in the value for "per cent air voids." Furthermore, the amount of water absorbed within the individual soil particles has been assumed sometimes to be located entirely within the intergranular space between the particles and to be available to influence values for "per cent air voids" and "degree of saturation," that is, the absorbed water is not subtracted from the total water content when calculating "degree of saturation" and "per cent air voids."

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[3] Standard Method of Test for Specific Gravity of Soils (D 854), 1955 Book of ASTM Standards, Part 3, p. 1786.

[4] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1395.

[5] Tentative Definitions of Terms and Symbols Relating to Soil Mechanics (D 653 - 42 T) 1955 Book of ASTM Standards, Part 3, p. 1825.

Specialists in soil mechanics clearly recognize the significance of the various factors that influence the values calculated for the void properties of a soil mass. However, since this does not always seem to be the case in routine soil engineering, it is one of the principal objectives of this paper to point out the magnitude of the errors in the values for void properties that can result from using the wrong specific gravity of the soil and from failure to take into account the portion of the water that has been absorbed by the water-permeable porosity of the individual soil particles.

As will be shown later, these errors tend to be relatively small for either saturated or unsaturated fine-textured soils with high moisture contents or relatively low bulk densities. On the other hand, they can be appreciable for coarse-grained soils such as sands and gravels. These errors can be large enough that difficulty can arise when trying to relate the values for void properties with the actual field performance of soils and even when checking the validity of a proposed theory or equation by means of laboratory tests, for example, permeability.

For this paper, it is assumed that correct values for the void properties of soils will be obtained if they are calculated by

(*a*) employing the ASTM bulk specific gravity of the soil to determine the volume of the solid particles in a given soil mass;

(*b*) subtracting the absorbed water from the total water content to find the "effective" quantity of water available to influence values for "per cent air voids" and "degree of saturation."

These assumptions appear to be justified by the following considerations:

1. Water that has been absorbed into a soil particle (*c* of Fig. 1, and cross-hatched area of Fig. 3) is lost insofar as its influence on most engineering properties of the soil are concerned. If "effective moisture content" is defined as the total moisture content of the soil (oven-dry basis) minus the absorbed water, it is clear that only the "effective moisture content" is available to (*a*) act as a cementing or lubricating medium, (*b*) contribute to gain or loss in shear strength, (*c*) drain during a consolidation test, (*d*) provide the soil with plasticity, workability, and ease of compaction during construction, and (*e*) serve as a void filling material in the intergranular space between the soil particles. Consequently, recognition of this "effective moisture content" is important regardless of what specific gravity is employed to determine the volume of the solid particles in a given soil mass.

2. As pointed out earlier, two different specific gravities may be measured for coarser soils: ASTM bulk, and ASTM apparent specific gravity. In routine soil engineering, the significance of the

difference between these two specific gravities is frequently overlooked. If ASTM apparent specific gravity has been specified, it is often used to calculate the volume of the solid particles in a given soil mass in precisely the same way that ASTM bulk specific gravity is employed for this purpose. When used in this manner, the volume of the solid particles in a given soil mass when calculated by means of the two specific gravities can be substantially different.

The internal skeleton or framework of any given soil mass consists of the volume of the soil particles calculated on the basis of their ASTM bulk specific gravity. This is illustrated by the full circles in Fig. 4. As ASTM apparent specific gravity is very often used, it results in an assumed shrinkage of each soil particle to the volume illustrated by the intermediate full circles in Fig. 4. The assumed shrinkage of each particle is equal to the volume of its water-permeable pore space after immersion for 24 hr, Fig. 4. For example, with 2 per cent water absorption and an ASTM bulk specific gravity of 2.50, the use of ASTM apparent instead of ASTM bulk specific gravity results in a volume shrinkage of 5 per cent for each particle. This shrinkage in volume has not actually occurred but it illustrates the degree of error to which ASTM apparent specific gravity, as frequently used, can lead when calculating the volume of the internal framework provided by the particles of a soil mass. An accurate knowledge of the actual volume of this framework of solid particles frequently facilitates better understanding of other measured properties of the soil mass.

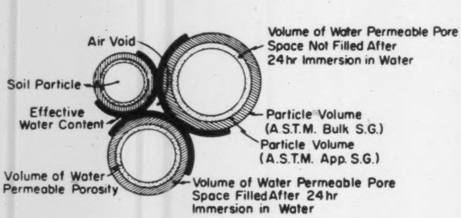
Air Void
Soil Particle
Effective Water Content
Volume of Water Permeable Porosity
Volume of Water Permeable Pore Space Not Filled After 24 hr Immersion in Water
Particle Volume (A.S.T.M. Bulk S.G.)
Particle Volume (A.S.T.M. App. S.G.)
Volume of Water Permeable Pore Space Filled After 24 hr Immersion in Water

Fig. 4.—Illustrating the Influence of Water Absorption and ASTM Bulk and Apparent Specific Gravities on Values Calculated for the Void Properties of Soils.

Sample Calculations

The magnitude of the errors that can result from calculating values for the void properties of a soil mass by methods frequently used will be illustrated by four numerical examples. These errors are due to,

1. Failure to allow for water absorbed into the water-permeable porosity of the individual soil particles.

2. Using ASTM apparent instead of ASTM bulk specific gravity to determine the volume occupied by the solid particles in the soil mass.

In each of the four examples, what are considered to be correct values for the void properties will also be presented, based upon ASTM bulk specific gravity of the soil particles and making full allowance for water absoprtion.

The first three examples pertain to soils with the relatively high water absorption value of 2.1 lb of water per 100 lb of oven-dry soil. The first example is concerned with a well-compacted, dense-graded gravel for which normal void values are obtained. The second example is based on the same material but illustrates the case of negative air voids and a degree of saturation greater than 100 per cent. The third example considers a fine-textured soil at quite high moisture content.

In contrast to the first example, the fourth example is concerned with a well-compacted, dense-graded gravel having the relatively low water absorption value of 0.6 lb of water per 100 lb of oven-dry soil.

Values for void properties associated with these four sets of conditions are

TABLE I.—EVALUATING THE VOID PROPERTIES OF WELL-COMPACTED, DENSE-GRADED GRAVEL HAVING MODERATE WATER ABSORPTION.

Water absorption of soil particles, per cent	2.1
Bulk specific gravity of the soil mass	2.26
Moisture content of the soil mass, per cent	6.8

Method	Specific Gravity of Soil Particles		Allowance for Water Absorption	Inter-Granular Voids, per cent	Void Ratio	Air Voids, per cent	Saturation, per cent
A	ASTM bulk	2.54	No	16.7	0.200	2.3	86.2
B	ASTM apparent	2.68	No	21.1	0.267	6.7	68.2
C	ASTM bulk	2.54	Yes	16.7	0.200	6.7	59.9

TABLE II.—ILLUSTRATING THE CASE OF NEGATIVE AIR VOIDS, WHY IT OCCURS, AND HOW IT IS AVOIDED. (WELL-COMPACTED, DENSE-GRADED GRAVEL WITH MODERATE WATER ABSORPTION.)

Water absorption of soil particles, per cent	2.1
Bulk specific gravity of the soil mass	2.32
Moisture content of the soil mass, per cent	8.3

Method	Specific Gravity of Soil Particles		Allowance for Water Absorption	Inter-Granular Voids, per cent	Void Ratio	Air Voids, per cent	Saturation, per cent
A	ASTM bulk	2.54	No	15.7	0.186	−2.1	113.4
B	ASTM apparent	2.68	No	20.1	0.252	2.3	88.6
C	ASTM bulk	2.54	Yes	15.7	0.186	2.3	84.7

TABLE III.—EVALUATING THE VOID PROPERTIES OF A FINE-TEXTURED SOIL AT RELATIVELY HIGH MOISTURE CONTENT. (SOIL WITH MODERATE WATER ABSORPTION.)

Water absorption of soil particles, per cent	2.1
Bulk specific gravity of the soil mass	1.60
Moisture content of the soil mass, per cent	50.0

Method	Specific Gravity of Soil Particles		Allowance for Water Absorption	Inter-Granular Voids, per cent	Void Ratio	Air Voids, per cent	Saturation, per cent
A	ASTM bulk	2.54	No	58.0	1.38	4.7	91.9
B	ASTM apparent	2.68	No	60.2	1.51	6.9	88.5
C	ASTM bulk	2.54	Yes	58.0	1.38	6.9	88.1

TABLE IV.—EVALUATING THE VOID PROPERTIES OF A WELL-COMPACTED, DENSE-GRADED GRAVEL HAVING RELATIVELY LOW WATER ABSORPTION.

Water absorption of soil particles, per cent	0.6
Bulk specific gravity of the soil mass	2.26
Moisture content of the soil mass, per cent	6.8

Method	Specific Gravity of Soil Particles		Allowance for Water Absorption	Inter-Granular Voids, per cent	Void Ratio	Air Voids, per cent	Saturation, per cent
A	ASTM bulk	2.54	No	16.7	0.200	2.3	86.2
B	ASTM apparent	2.58	No	18.0	0.220	3.6	80.0
C	ASTM bulk	2.54	Yes	16.7	0.200	3.6	78.4

listed in Tables I, II, III, and IV. Methods A and B referred to in each of the four tables, although in fairly common use, are believed to provide erroneous values for certain void properties of a soil mass. Method A is in error with respect to "per cent air voids" and "per cent saturation" because it makes no allowance for water absorption into individual soil particles. Method B provides incorrect values for "per cent intergranular voids," "void ratio," and "per cent saturation" because the volume occupied by the soil particles is too low when calculated by their ASTM apparent specific gravity. The values for void properties given by method C are considered to be correct because full allowance is made for the water absorbed by the individual soil particles and the volume of the soil particles is calculated on the basis of their ASTM bulk specific gravity.

The procedure employed for calculating values of void properties by means of methods A and B in each of the four tables is illustrated by the following example, using data from Table I. In all cases the specific gravity of water is assumed to be 1.000.

Basis of Computation—100 cu cm of the soil mass.

Weight of 100 cu cm of the soil mass = 226 g.

Weight of water in 100 cu cm of the soil mass $= \left(\frac{6.8}{106.8}\right)(226) = 14.4$ g.

Weight of soil in 100 cu cm of the soil mass $= \left(\frac{100}{106.8}\right)(226) = 211.6$ g.

Volume of water in 100 cu cm of the soil mass = 14.4 cu cm.

Volume of soil in 100 cu cm of the soil mass

Based on ASTM bulk specific gravity $= \frac{211.6}{2.54} = 83.3$ cu cm.

Based on ASTM apparent specific gravity $= \frac{211.6}{2.68} = 78.9$ cu cm.

Therefore, the values for the various void properties of this soil mass as given by Methods A and B are:

	Method A	Method B
Intergranular voids, per cent	$100 - 83.3 = 16.7$	$100 - 78.9 = 21.1$
Void ratio	$\frac{16.7}{83.3} = 0.200$	$\frac{21.1}{78.9} = 0.267$
Air voids, per cent	$100 - (83.3 + 14.4) = 2.3$	$100 - (78.9 + 14.4) = 6.7$
Saturation, per cent	$\left(\frac{14.4}{16.7}\right)(100) = 86.2$	$\left(\frac{14.4}{21.1}\right)(100) = 68.2$

To obtain what are believed to be correct values for the void properties listed opposite method C in each of the four tables, the procedure illustrated by the following computation based on the data of Table I was employed:

Basis of Computation—100 cu cm of the soil mass

Weight of 100 cu cm of the soil mass = (100) (2.26) = 226 g.

Weight of water in 100 cu cm of the soil mass $= \left(\frac{6.8}{106.8}\right)(226) = 14.4$ g.

Weight of soil in 100 cu cm of the soil mass $= \left(\frac{100}{106.8}\right)(226) = 211.6$ g.

Volume of water in 100 cu cm of the soil mass = 14.4 cu cm.

Volume of absorbed water in 100 cu cm of the soil mass $= \left(\frac{2.1}{100}\right)(211.6) = 4.4$ cu cm.

Volume of unabsorbed ("effective") water = 14.4 − 4.4 = 10.0 cu cm.

Volume of soil in 100 cu cm of the soil mass

Based on ASTM bulk specific gravity $= \frac{211.6}{2.54} = 83.3$ cu cm.

Consequently, the correct values for the void properties of this soil as given by Method C are:

$$\text{Intergranular voids, per cent} = 100 - 83.3 = 16.7$$

$$\text{Void ratio} = \frac{16.7}{83.3} = 0.200$$

$$\text{Air voids, per cent} = 100 - (83.3 + 10.0) = 6.7$$

$$\text{Saturation, per cent} = \frac{(10.0)}{(16.7)}\,(100) = 59.9.$$

Example No. 1

Of the eight void values listed for methods A and B in Table I, only three are correct. Opposite method A, the values for "per cent intergranular voids," 16.7, and for "void ratio," 0.200, are correct, but the values for "per cent air voids," 2.3, and for "per cent saturation," 86.2, are in error. The errors are due to failure to allow for the 2.1 per cent of water absorption by the soil particles. Opposite method B, the value for "per cent air voids," 6.7, is correct, but the values for "per cent intergranular voids," 21.1, for "void ratio," 0.267, and for "per cent saturation," 68.2, are erroneous. In this case, the errors are caused by the use of ASTM apparent specific gravity to calculate the volume occupied by the soil particles.

Table I indicates the void ratio of 0.267 calculated on the basis of ASTM apparent specific gravity to be 33.5 per cent greater than the void ratio of 0.200 provided by ASTM bulk specific gravity. Taylor (**3**) suggests that under certain conditions the permeability of a soil varies approximately with the square of its void ratio. In this particular case, if the void ratio were calculated on the basis of ASTM apparent instead of ASTM bulk specific gravity, the anticipated permeability of the soil given by Taylor's equation would be about 78 per cent too high and could be a major cause of very poor agreement with the actually measured value for permeability.

Method C in Table I is believed to provide correct values for each of the void properties listed. These are seen to be quite different in several cases from those listed opposite methods A and B. It should be particularly noted that the correct value of 59.9 per cent saturation is much less than the per cent saturation values given by methods A and B.

The comments that have just been made concerning Table I apply in greater or less degree to the data of Tables II, III, and IV as well.

Example No. 2

The most interesting items of Table II are the negative value of −2.1 for "per cent air voids" and the value of 113.4 for "per cent saturation" listed opposite method A. These impossible values result because no allowance was made for the 2.1 per cent of water absorbed by the water-permeable pore space within the individual aggregate particles. They occurred because it was assumed that the total moisture content was available to fill the intergranular void space between the aggregate particles. When allowance is made for the 2.1 per cent of water absorbed by the soil particles, the correct values for "per cent air voids," 2.3, and for "per cent saturation," 84.7, listed opposite method C are obtained.

Example No. 3

The data of Table III indicate that differences in void values due to using either the ASTM bulk or ASTM apparent specific gravity of the soil and to either allowing for or neglecting water absorption are much smaller for a fine-textured soil having a relatively high moisture content than for a well-compacted, dense-graded coarse-grained soil (Tables I and II) when the specific gravity and water absorption values are the same for both soils. Generally speaking, the differences between the values listed for each void property in Table III opposite methods A, B, and C are approaching the range of experimental error.

Example No. 4

In contrast to Table I, the differences between values for each void property listed in Table IV for a well-compacted, dense-graded gravel having a relatively low water absorption, 0.6 per cent, tend to be rather small, regardless of whether methods A, B, or C are employed to calculate them. Nevertheless, the spread in values for "per cent saturation" is twice that of Table III, and the range in values for "void ratio" is about 11 per cent.

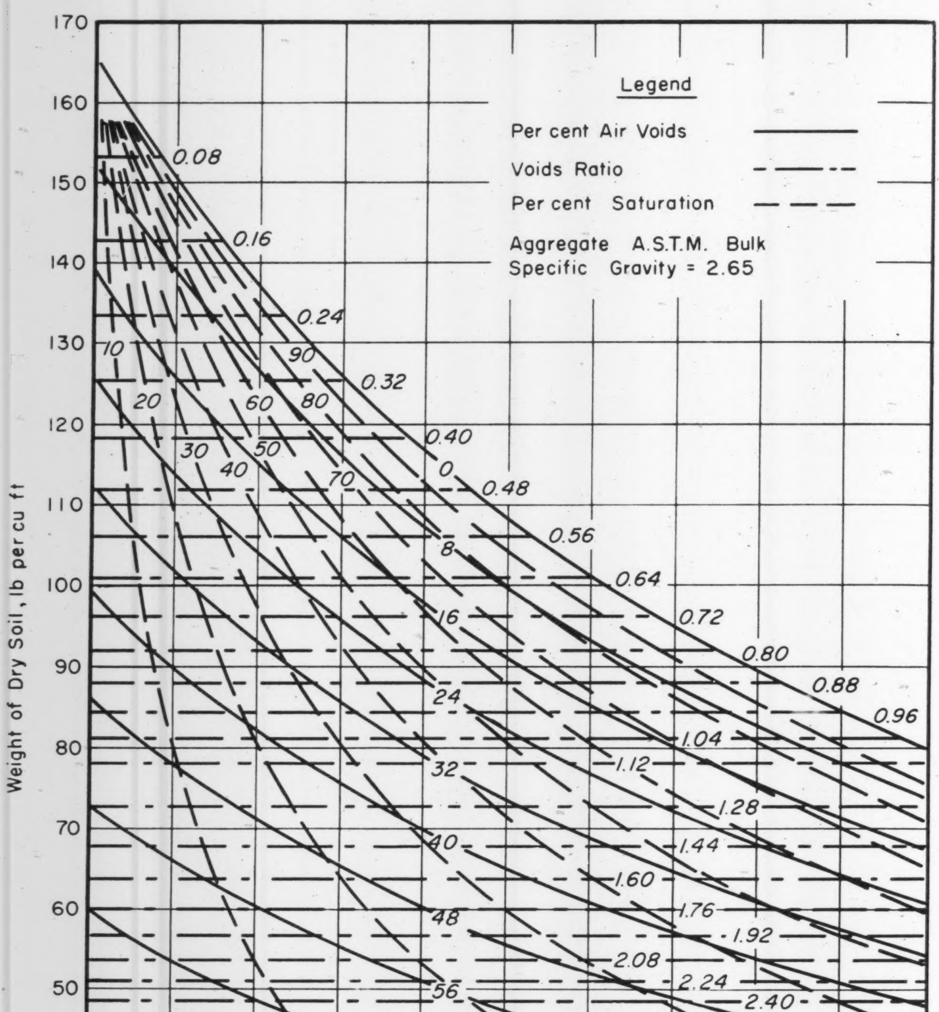

FIG. 5.—Relationships between Density, Moisture Content, Per Cent Saturation, Void Ratio and Air Voids for Soils (Dry Soil Basis).

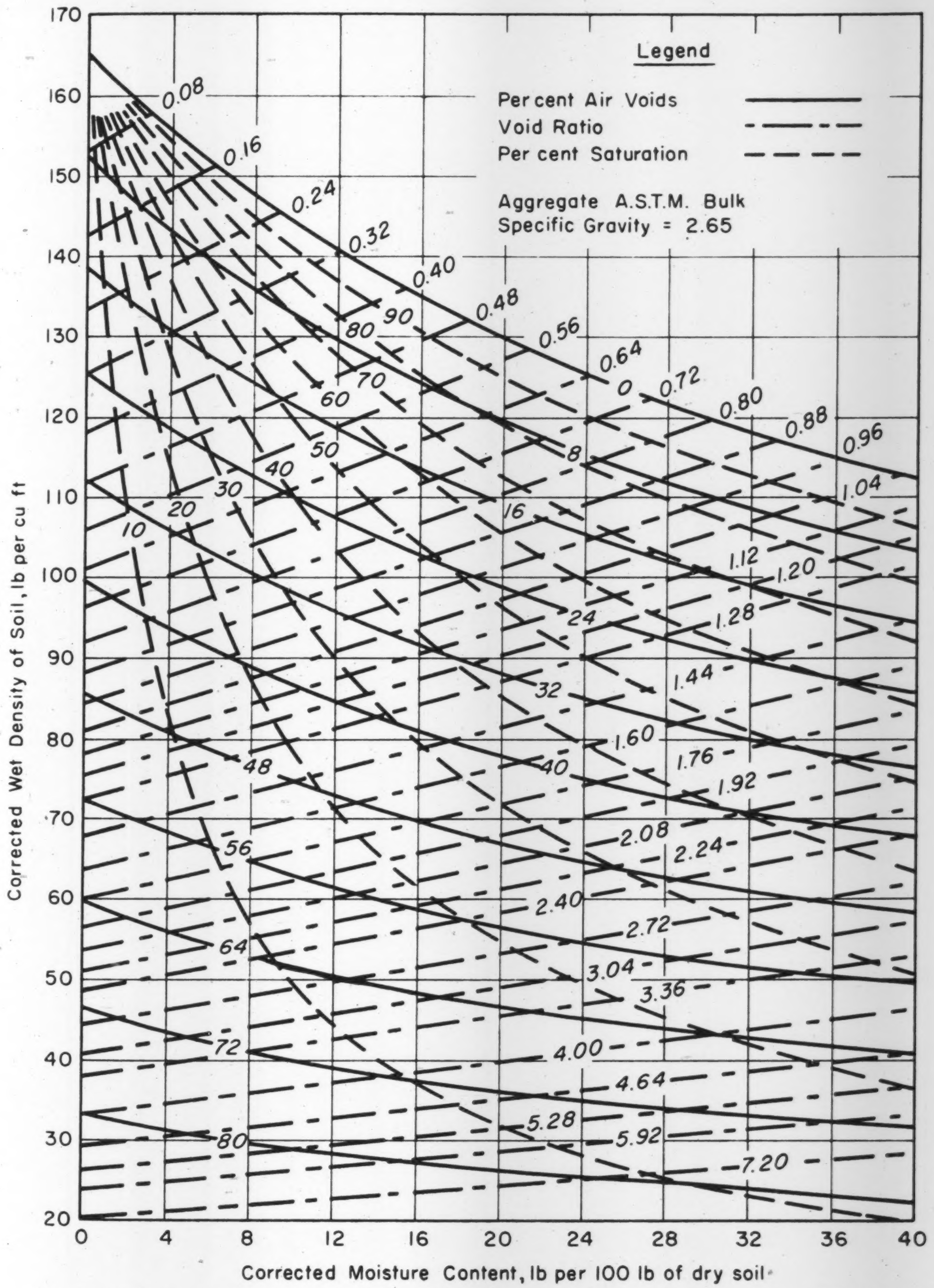

Fig. 6.—Relationships between Density, Moisture Content, Per Cent Saturation, Void Ratio and Air Voids for Soils (Wet Soil Basis).

To summarize rather briefly, the data contained in Tables I and II show that serious errors can occur if incorrect methods are used to calculate void values for well-compacted, dense-graded, coarse-textured soil masses when the water absorption of the individual soil particles is relatively high. These errors are sufficiently large that they could make it very difficult to relate void characteristics with measured values for engineering properties such as permeability, capillarity, drainage, strength, etc., of these soil masses. On the other hand, for fine-textured soils composed of particles with quite high water absorption and for coarse-textured soils having relatively low water absorption, Tables III and IV demonstrate that the differences in values for each void property are fairly small regardless of whether the erroneous or correct procedures represented by Methods A and B or C, respectively, are employed to calculate them.

Useful Charts for Evaluating Void Properties of Soils

When the water absorption and ASTM bulk specific gravity of the soil particles and the specific gravity of the water (or other liquid) are known, relationships must exist between five important properties of homogeneous soil masses as a matter of mathematical necessity. These five properties, illustrated in Figs. 5 and 6, are density of the soil mass in pounds per cubic foot, moisture content, void ratio, per cent air voids, and per cent saturation. The density of the soil mass and its moisture content are usually measured and have been made the ordinate and abscissa, respectively, of Figs. 5 and 6. On the basis of density and moisture content, the void ratio, per cent air voids, and per cent saturation of the soil mass can be calculated. Families of curves for each of these three variables are shown in Figs. 5 and 6. Figure 5 is based on the dry density of a soil mass, and Fig. 6 on its wet density.

Charts somewhat similar to those of Figs. 5 and 6 have been developed for bituminous paving mixtures (**4,5,6**).

Figures 5 and 6 can be used with precision only when the ASTM bulk specific gravity of the soil is 2.65 and when the specific gravity of the water or other fluid is 1.000. This is also true of Figs. 7, 8, and 10 to 14. If the specific gravity of either material, or of both, differs from these values, none of these figures can be used with accuracy. Consequently, similar charts should be prepared for each combination of specific gravities for the soil and water or other fluid under consideration. Since the specific gravity of water is very nearly unity over a fairly wide range of ordinary temperatures, it is usually necessary to consider only the ASTM bulk specific gravity of the soil.

Figures 5 and 6 are based in part upon the amount of soil moisture available to fill, or to partially fill, the intergranular space between the soil particles. In the case of absorptive soils, the quantity of water absorbed into the water-permeable porosity within the individual soil particles (*b* or *c* in Fig. 1) must be subtracted from the total water content of the sample (oven-dry basis). This gives the "corrected moisture content," or the quantity of water actually located in the intergranular space between the soil particles (the "effective" water content of Fig. 1), which is the abscissa of Figs. 5 and 6.

Figures 5 and 6 are also based upon the ASTM bulk specific gravity of the soil particles. For absorptive soils, the water that is absorbed by the individual soil particles must be taken into account. Otherwise the actual specific gravity of the particles is increased from ASTM bulk to some higher value of specific gravity that varies directly with the

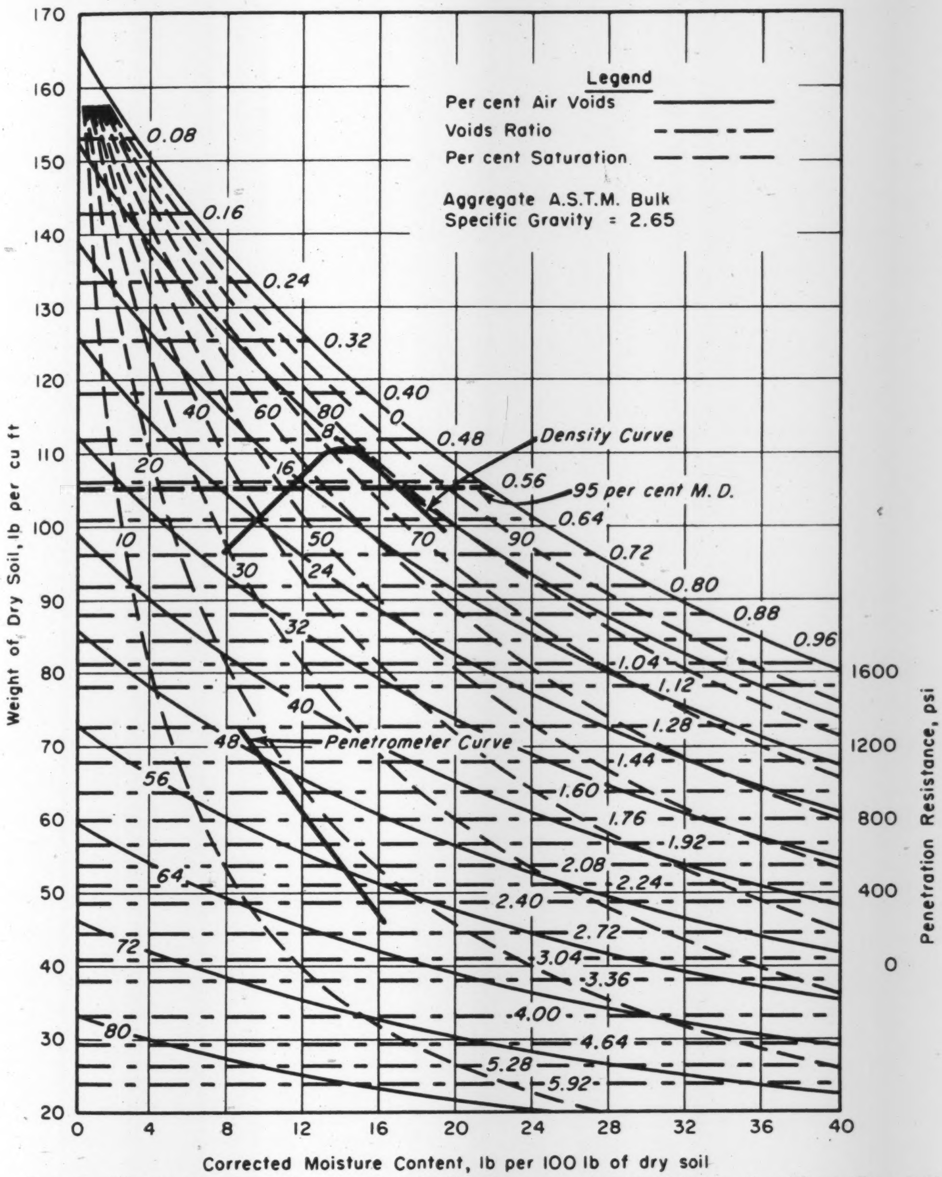

Fig. 7.—Illustrating Compaction and Stability Curves on the Void Properties Chart (Dry Soil Basis).

quantity of water absorbed up to the value of the ASTM bulk specific gravity saturated surface-dry basis[2] or slightly higher. Whether or not water absorption occurs has no influence on the ordinate of Fig. 5, which is the usual soil density on an oven-dry basis. On the other hand, to use Fig. 6 correctly, from the measured wet density of the soil mass, the weight of water absorbed within the

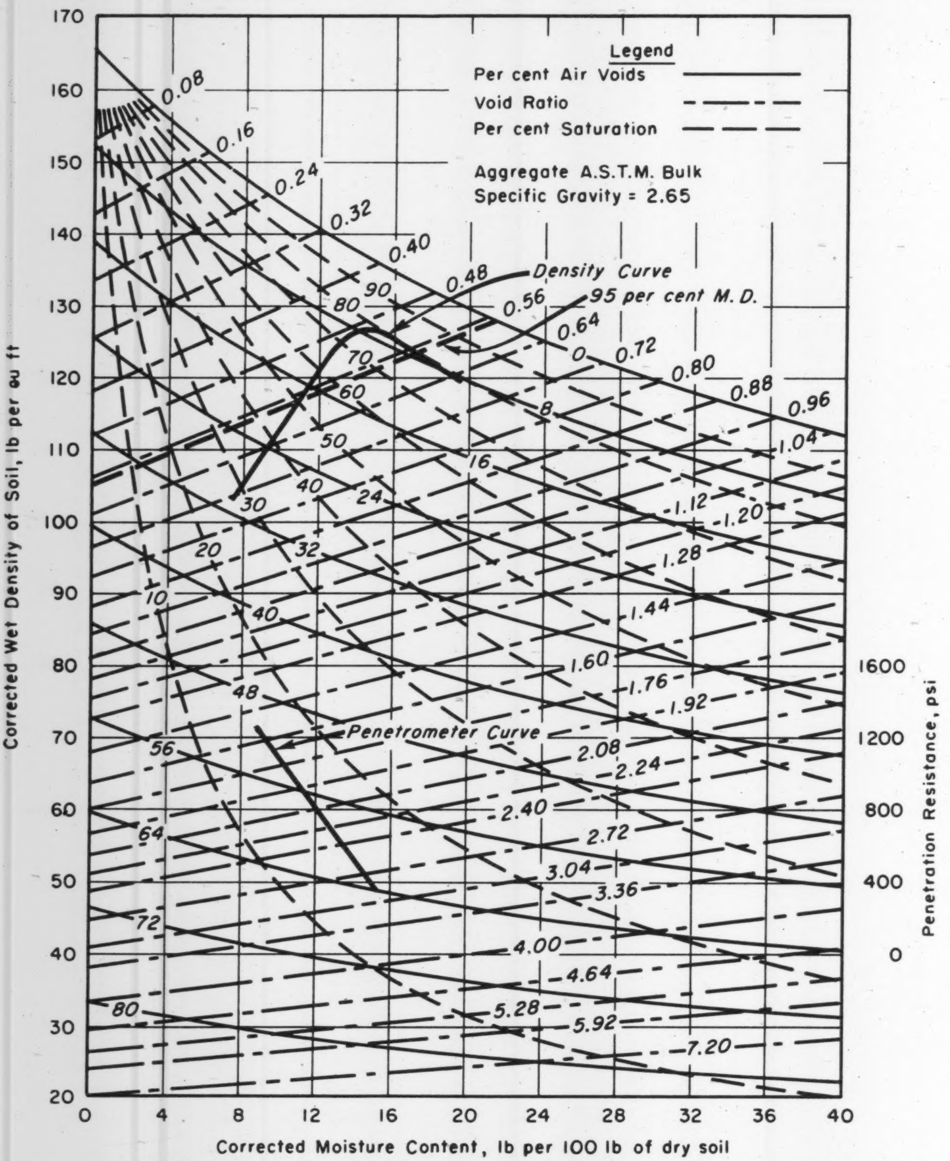

Fig. 8.—Illustrating Compaction and Stability Curves on the Void Properties Chart (Wet Soil Basis).

individual soil particles must be subtracted, giving the "corrected wet density" of the soil, which is the ordinate of Fig. 6.

As just indicated, Figs. 5 and 6 can be used to provide correct values for void properties regardless of the amount of water absorbed by the soil particles but only if the measured moisture content and measured wet density are corrected for the weight of absorbed water. This in effect corrects the conditions of Fig.

3 to those of Fig. 2. Consequently, Figs. 5 and 6 are based essentially on Fig. 2, or, expressed more precisely, they are based upon conditions represented by Fig. 3 (water absorption) that have been corrected to those of Fig. 2 (no water absorption).

Where absorptive soils are concerned, any attempt to use Figs. 5 and 6 on the basis of ASTM apparent specific gravity, measured wet density, or measured water content, will lead to values for "void ratio," "per cent saturation," and "per cent air voids," having the margin of error illustrated by the data calculated by methods A and B that are listed in Tables I to IV. The amount of error will be quite large for dense-graded, coarse-grained soils with rather high water absorption but much smaller for fine-textured soils having the same water absorption or for dense-graded, coarse-grained soils with relatively low water absorption.

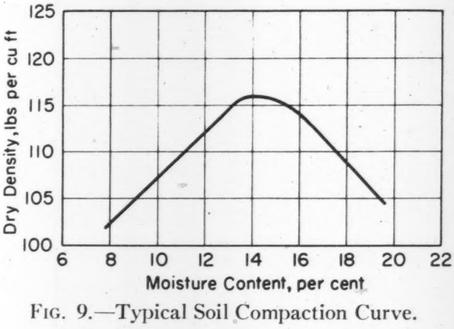

Fig. 9.—Typical Soil Compaction Curve.

If the amount of water absorbed by the individual soil particles is negligible or zero, no density or moisture corrections can be made. In this case, the measured density becomes the "corrected wet density of soil" of Fig. 6, and the measured total water content (oven-dry basis) becomes the "corrected moisture content" of Figs. 5 and 6.

Figure 7 is similar to Fig. 5, but a soil compaction curve from a standard compaction test and the corresponding strength curve provided by a Proctor needle have been added. Figure 8 is similar to Fig. 7, but the density of the soil is expressed on an oven-dry basis in Fig. 7 and on a corrected wet basis in Fig. 8.

The soil compaction and strength curves of Figs. 7 and 8 are similar to those usually plotted on the basis of measured dry or wet density *versus* measured moisture content. However, by plotting the *corrected* soil compaction curve on charts like those of Figs. 7 and 8, the investigator or engineer automatically obtains complete information concerning the void properties of a soil, without any need for the usual computations required to evaluate these factors. For example, the point on the compaction curve in Fig. 7 representing a corrected moisture content of 13.0 per cent and an oven-dry density of 109.8 lb per cu ft, indicates by interpolation:

Void ratio	0.51
Air voids, per cent	10.7
Saturation, per cent	68.5

Identical soil compaction data are represented by the compaction curves of both Fig. 7 and Fig. 8, the curve of Fig. 7 being on a dry density basis while that of Fig. 8 is based on "corrected wet density." Furthermore, it will be seen that for a corrected moisture content of 13.0 per cent, the compaction curve of Fig. 8 gives the same values for "void ratio," "per cent air voids," and "per cent saturation," that have just been listed for the compaction curve of Fig. 7 for this moisture content. This must be the case of course, if both diagrams are equivalent, one based on oven-dry and the other on "corrected wet" density.

The usual compaction curve for measured density *versus* measured moisture content tells nothing of the void charac-

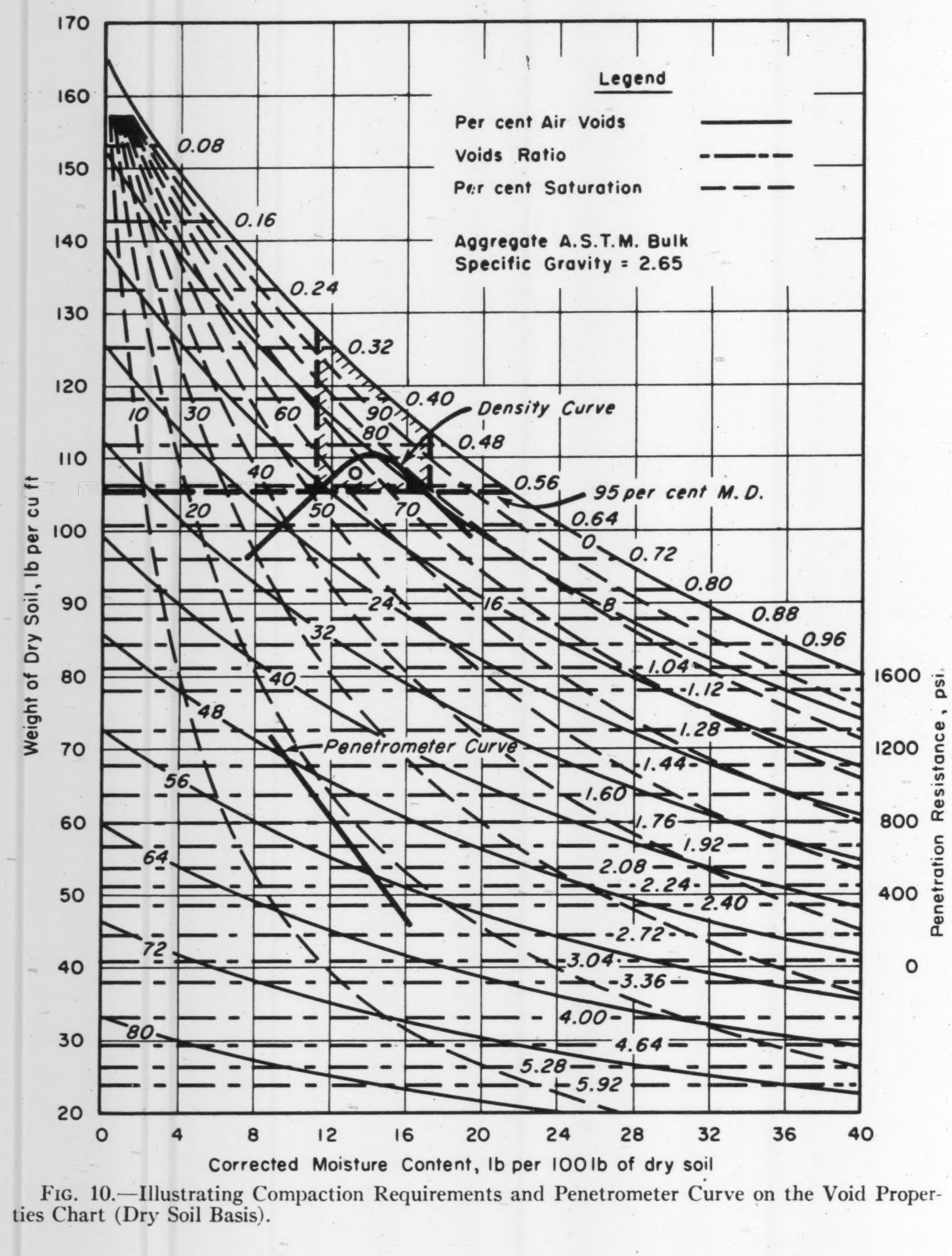

Fig. 10.—Illustrating Compaction Requirements and Penetrometer Curve on the Void Properties Chart (Dry Soil Basis).

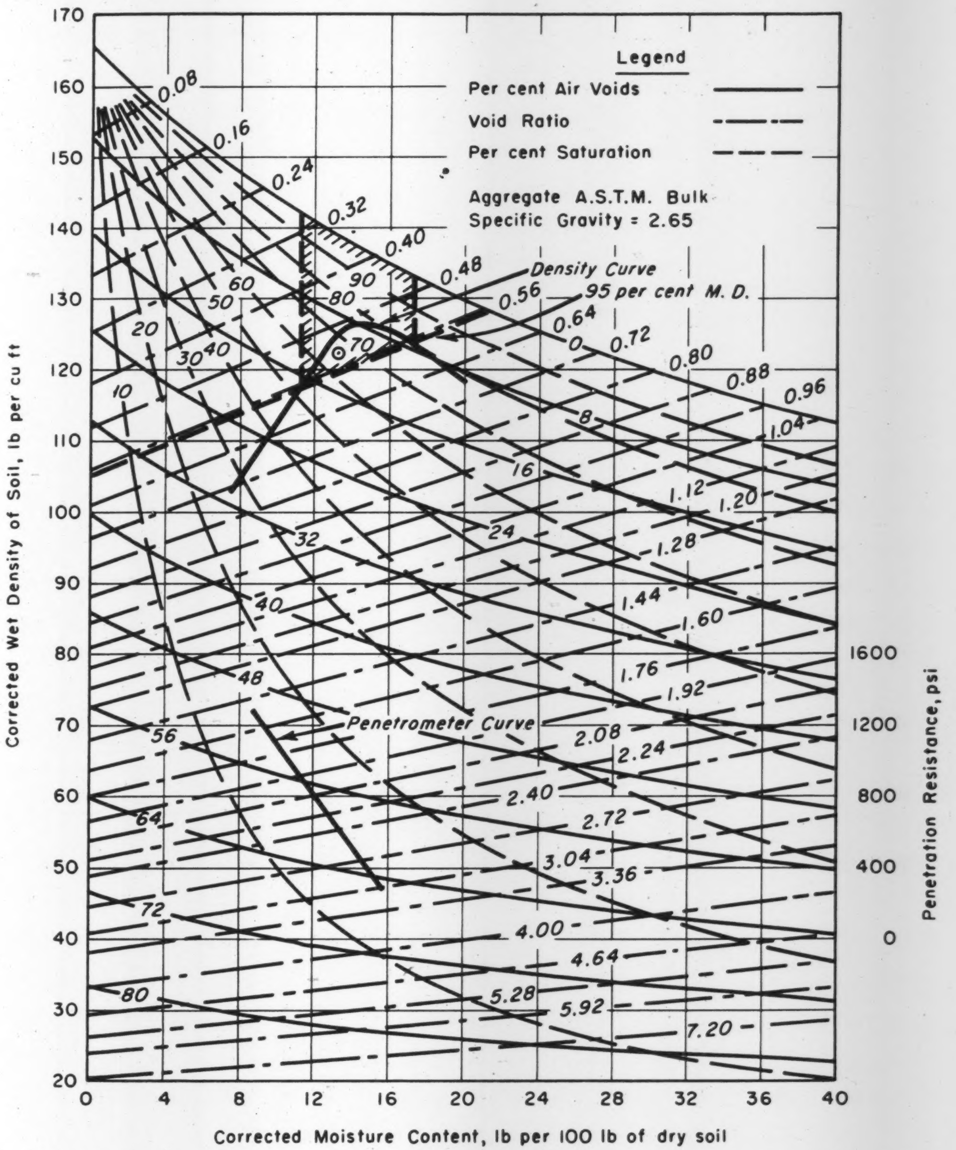

Fig. 11.—Illustrating Compaction Requirements and Penetrometer Curve on the Void Properties Chart (Wet Soil Basis).

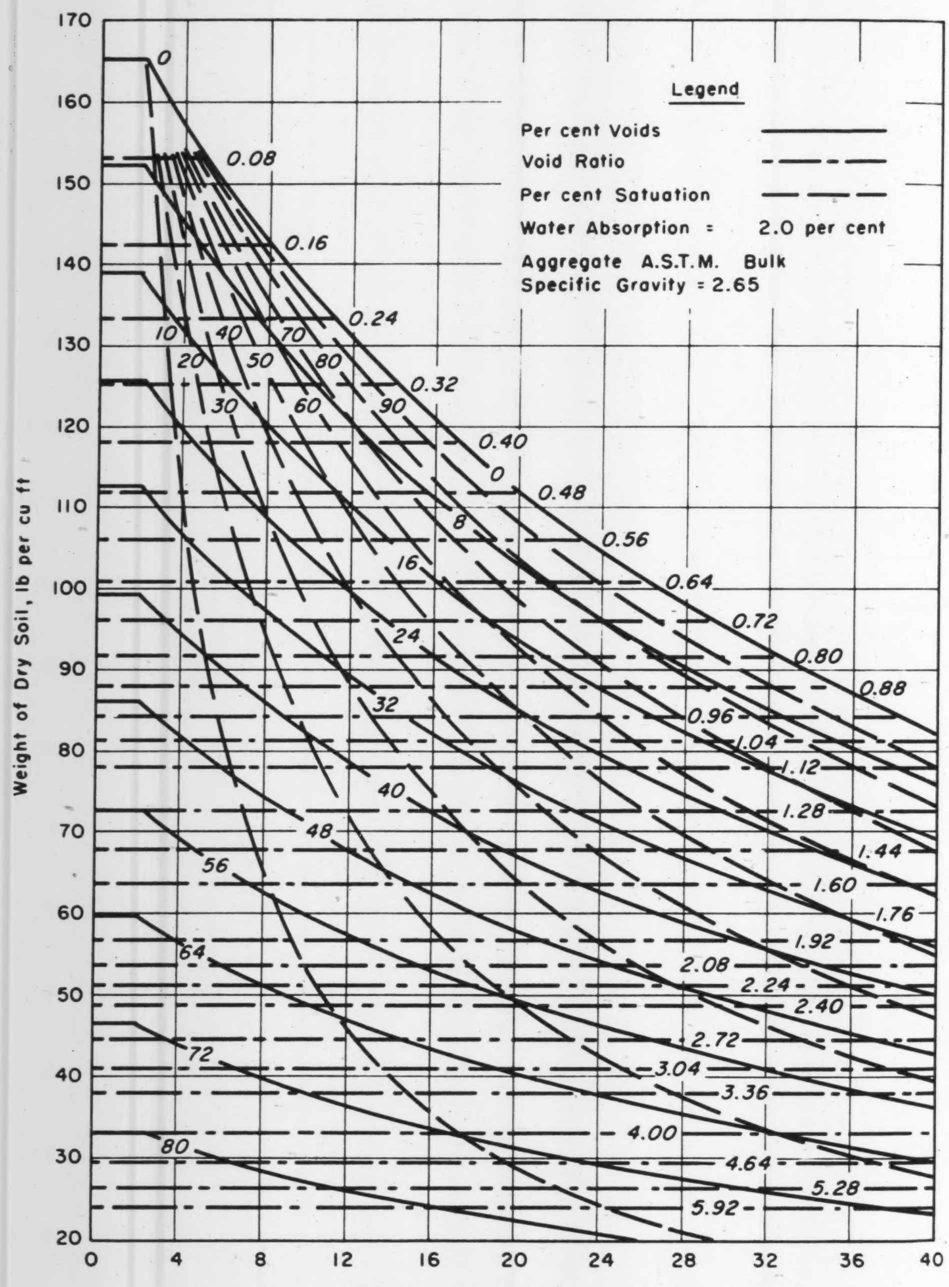

Fig. 12.—Relationships between Density, Moisture Content, per cent Saturation, Void Ratio and Air Voids for Soils with 2 per cent Water Absorption (Dry Soil Basis).

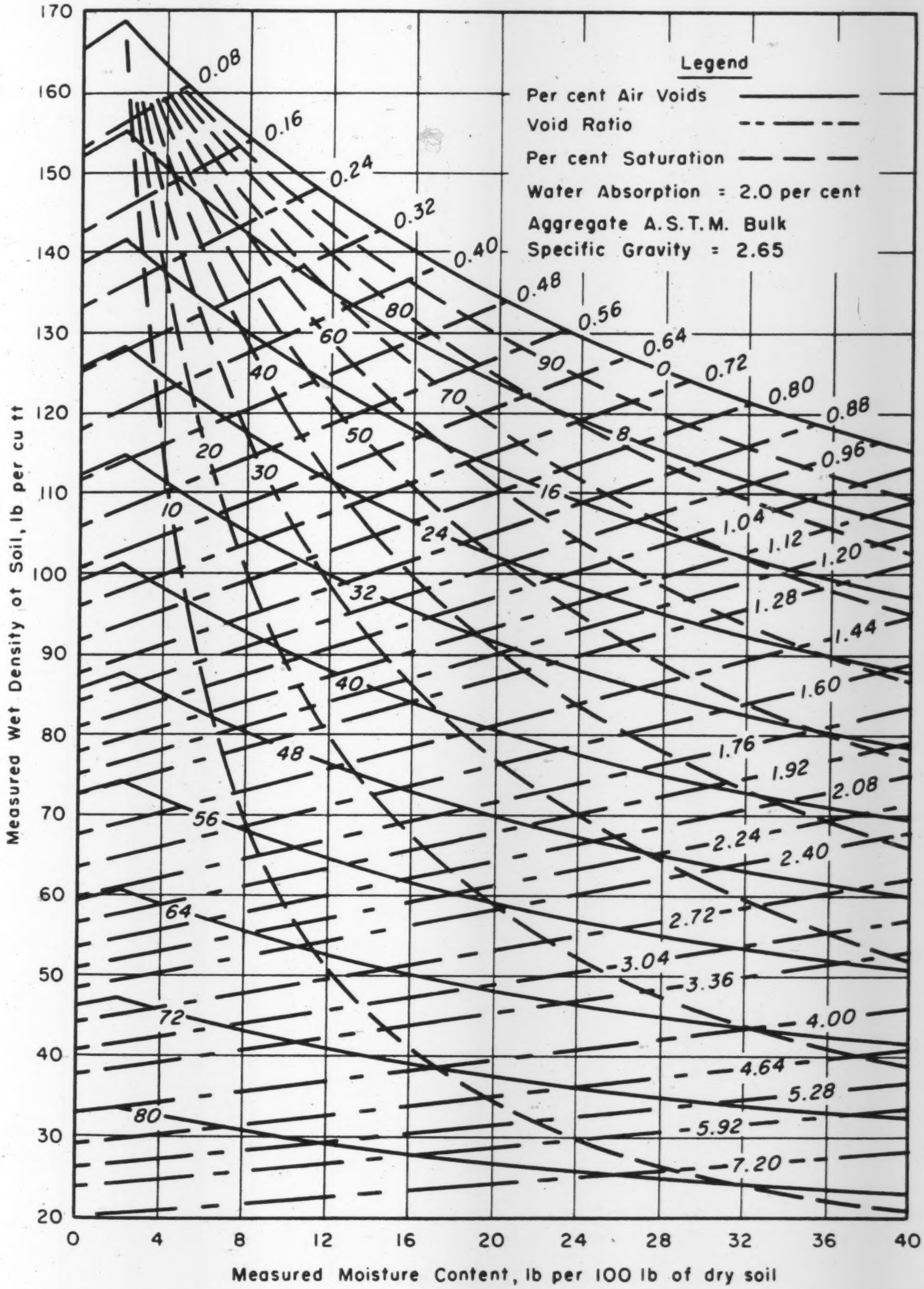

Fig. 13.—Relationships between Density, Moisture Content, per cent Saturation, Void Ratio and Air Voids for Soils with 2 per cent Water Absorption (Wet Soil Basis).

teristics of the soil, and very often the graph does not even show the zero air voids line, Fig. 9. On the other hand, by merely plotting the corrected compaction curve on Fig. 7 or Fig. 8, accurate values for the void properties of the soil are provided by visual inspection and without the usual expenditure of time and effort needed for calculating them.

Figures 7 and 8 are useful for general soil analysis quite apart from soil compaction. They can be used to furnish void values for a representative sample from either an undisturbed or disturbed soil mass. As soon as the point representing the corrected density and corrected moisture content of the soil sample has been plotted on Fig. 7 or Fig. 8 or on a similar diagram, this automatically enables values for "void ratio," "per cent air voids," and "per cent saturation" to be read directly from the chart.

By plotting moisture and density values for soils on charts like Figs. 7 and 8, certain unreasonable data may be detected immediately. This would avoid the situation that sometimes develops where, as ordinarily used, the measured moisture and density values imply negative air voids, per cent saturation greater than 100, etc. This is occasionally due to experimental error but is not infrequently caused by incorrect interpretation of data, as illustrated by Table II.

Specifications for earthwork often require that a soil be compacted to 95 per cent or to some other percentage of the maximum density given by a specified method of laboratory compaction. In Figs. 7 and 8, the lines corresponding to 95 per cent of maximum density have been drawn. The 95 per cent compaction line is horizontal in Fig. 7 but has a positive slope in Fig. 8. This line corresponds to a void ratio of 0.57 in both figures, and its intersection with the compaction curve in each diagram provides the same range of corrected moisture content, 11.2 to 17.2 per cent. This is to be expected, since the compaction curves of Figs. 7 and 8 are based upon the same basic moisture and density data. Figures 7 and 8 illustrate the somewhat different appearance of the two soil compaction diagrams when one is based on dry and the other on wet density.

Field Control of Soil Compaction

The enclosed areas on Figs. 10 and 11 indicate where all points representing field density tests should lie if the minimum compaction requirement is 95 per cent of maximum density and if the moisture content is to be maintained within the range of 11.2 to 17.2 per cent during the actual construction of an earthwork project using the soil represented by the compaction curve. In both figures, the bottom boundary of this area is the line representing 95 per cent of maximum density, the left-hand and right-hand boundaries indicate moisture contents of 11.2 and 17.2 per cent, respectively, and the top boundary is the zero air voids curve.

The use of Figs. 10 and 11 for controlling soil compaction in the field, will be illustrated by an example.

Example No. 5

1. *Test Data*

(*a*) ASTM bulk specific gravity of the soil, 2.65.

(*b*) Water absorption by the soil particles, per cent, 2.3.

(*c*) Specific gravity of water, 1.000.

(*d*) Measured density of field compacted soil, lb per cu ft, 124.7.

(*e*) Measured moisture content of field compacted soil, per cent, 15.5.

2. *Problem*

The problem consists of determining whether or not this sample from the field compacted layer of soil meets the compaction requirements represented by the enclosed areas of Figs. 10 and 11.

3. *Solution*

Basis of computation—1 cu ft of field compacted soil.

(*a*) Since the measured total moisture content of the soil is 15.5 per cent, and the water absorption into the individual soil particles is 2.3 per cent, the "corrected moisture content" of the soil sample is 15.5 − 2.3 = 13.2 per cent.

(*b*) Weight of 1 cu ft of the field compacted soil = 124.7 lb. Weight of 1 cu ft of the field compacted soil, oven-dry basis = $\left(\frac{100}{115.5}\right)$ (124.7) = 108 lb. Weight of water absorbed into soil particles in 1 cu ft of the field compacted soil = $\left(\frac{2.3}{100}\right)$ (108) = 2.5 lb. Therefore, the "corrected wet density" of 1 cu ft of the field compacted soil = 124.7 − 2.5 = 122.2 lb.

(*c*) Consequently, for Fig. 10, Weight of dry soil, lb per cu ft = 108. "Corrected moisture content," per cent = 13.2, and for Fig. 11, "Corrected wet density of soil," lb per cu ft = 122.2. "Corrected moisture content," per cent = 13.2.

(*d*) Plot the points representing these test data on Figs. 10 and 11 respectively (see small circles). Since these small circles lie within the area of each diagram that represents the specified compaction requirements for the soil, the layer has been rolled adequately at the point where the soil density and moisture content were measured.

Incidentally, it may be noted that the points represented by the small circles in Figs. 10 and 11, which were determined on the basis of the measured values for moisture content and density, automatically provide the following values for "void ratio," "per cent air voids," and "per cent saturation,"

Void ratio	0.535
Air voids, per cent	11.8
Saturation, per cent	66.0

The area representing acceptable moisture and density limits may often be narrower than those illustrated in Figs. 10 and 11. Sometimes the field moisture is maintained on the dry side of optimum moisture, and for other projects the wet side of optimum may be specified. It is a simple matter to establish areas on charts like those of Figs. 10 and 11 for which the width represents whatever range of moisture content the design requirements call for.

It should be emphasized that Figs. 10 and 11 can be used with accuracy only when the ASTM bulk specific gravity of the soil is 2.65. For soils with ASTM bulk specific gravities different from 2.65, similar charts based upon the actual ASTM bulk specific gravity of the soil should be prepared.

CHARTS FOR SPECIFIC VALUES OF WATER ABSORPTION

Figures 5, 6, 7, 8, 10, and 11 require that the measured moisture content of the soil be corrected for the water absorbed into the individual soil particles to give the "corrected moisture content." For Figs. 6, 8, and 11, the measured wet density of the soil must be corrected for the weight of water absorbed into the individual soil particles to provide the "corrected wet density." These charts have the particular merit that they provide correct values for "void ratio," "per cent air voids," and "per cent saturation," regardless of the quantity of water absorbed by the individual particles of soil.

On some large project, the quantity of water absorbed by the individual soil particles may be quite constant, for example, 2.0 per cent. In this case, it happens that a special chart can be employed, Figs. 12 or 13, for which the usual corrections to measured moisture content and measured density are not necessary. The abscissa of Figs. 12 and 13 is measured moisture content, and the ordinate is dry and measured wet density, respectively. Adjustment of measured moisture content and measured wet density for the 2.0 per cent of water absorbed by the soil particles is not required for Figs. 12 or 13 because the necessary correction has been incorporated into the construction of these charts. It should be carefully observed that the use of Figs. 12 and 13 is restricted to the particular case where the individual soil particles absorb 2 per cent of water and the ASTM bulk specific gravity of the soil is 2.65. Similar charts can be prepared for other constant values of soil water absorption such as 0.5, 1.0, 1.5 per cent, etc., and for soils with other ASTM bulk specific gravities.

It will be noted that the left-hand side of Figs. 12 and 13 is different from that of Figs. 5, 6, 7, 8, 10, and 11. In the latter, the curves representing "per cent air voids" and "per cent saturation" are carried through without a break to the ordinate axis representing a "corrected moisture content" of zero. In Figs. 12 and 13 on the other hand, a marked discontinuity occurs in the "per cent air voids" curves at a measured moisture content of 2 per cent. In addition, in Fig. 12 all curves representing various values for "per cent saturation" come to a common point at the top of the left-hand side, corresponding to a dry density of 165.36 lb per cu ft and a measured moisture content of 2 per cent (1 cu ft of material having an ASTM bulk specific gravity of 2.65 weighs 165.36 lb). In Fig. 13, the "per cent saturation" curves come to a similar common point representing a measured wet density of 168.66 lb per cu ft and a measured moisture content of 2 per cent (one cu ft of material having an ASTM bulk specific gravity of 2.65 and a water absorption of 2 per cent weighs 168.66 lb at a moisture content of 2 per cent).

These peculiarities on the left-hand side of Figs. 12 and 13 occur because of the assumption that the first 2 per cent of water added to the soil, or the last 2 per cent of water to leave the soil, under equilibrium conditions, will occupy the water-permeable porosity of the individual soil particles. The internal capillaries that make up this water-permeable porosity will usually be of very small diameter and the capillary suction pressure on any available water will be very high until they are completely filled.

When dealing with fine-textured clay soils, such factors as heat of wetting, hydration, ionic atmospheres, osmosis, Donnan equilibrium, solvation of ions, etc., make it difficult to determine exactly how very low moisture contents are distributed over and within an individual soil particle. For coarse-grained soils on the other hand, under equilibrium conditions, it seems reasonable to expect that the very large suction forces of the fine capillaries within each particle will tend to absorb added water, up to the water absorption value for the soil.

In any case, how to handle the factors that lead to these special features on the left-hand side of the diagram is a practical problem in soil engineering that is frequently overlooked but which is brought sharply into focus when diagrams like those of Figs. 12 and 13 are to be prepared.

Charts for Saturated Soils

For saturated soils, the "per cent air voids" is zero and the "per cent saturation" is 100. However, values of "void ratio" for masses of saturated soils may vary over wide limits depending upon the specific gravity of the soil, and its moisture content.

Figure 14, which is restricted to saturated soils, provides zero air voids curves for soils with ASTM bulk specific gravities ranging from 2.52 to 3.00. Successive zero air voids curves differ by 0.02 unit of ASTM bulk specific gravity. Figure 14 can be used in three

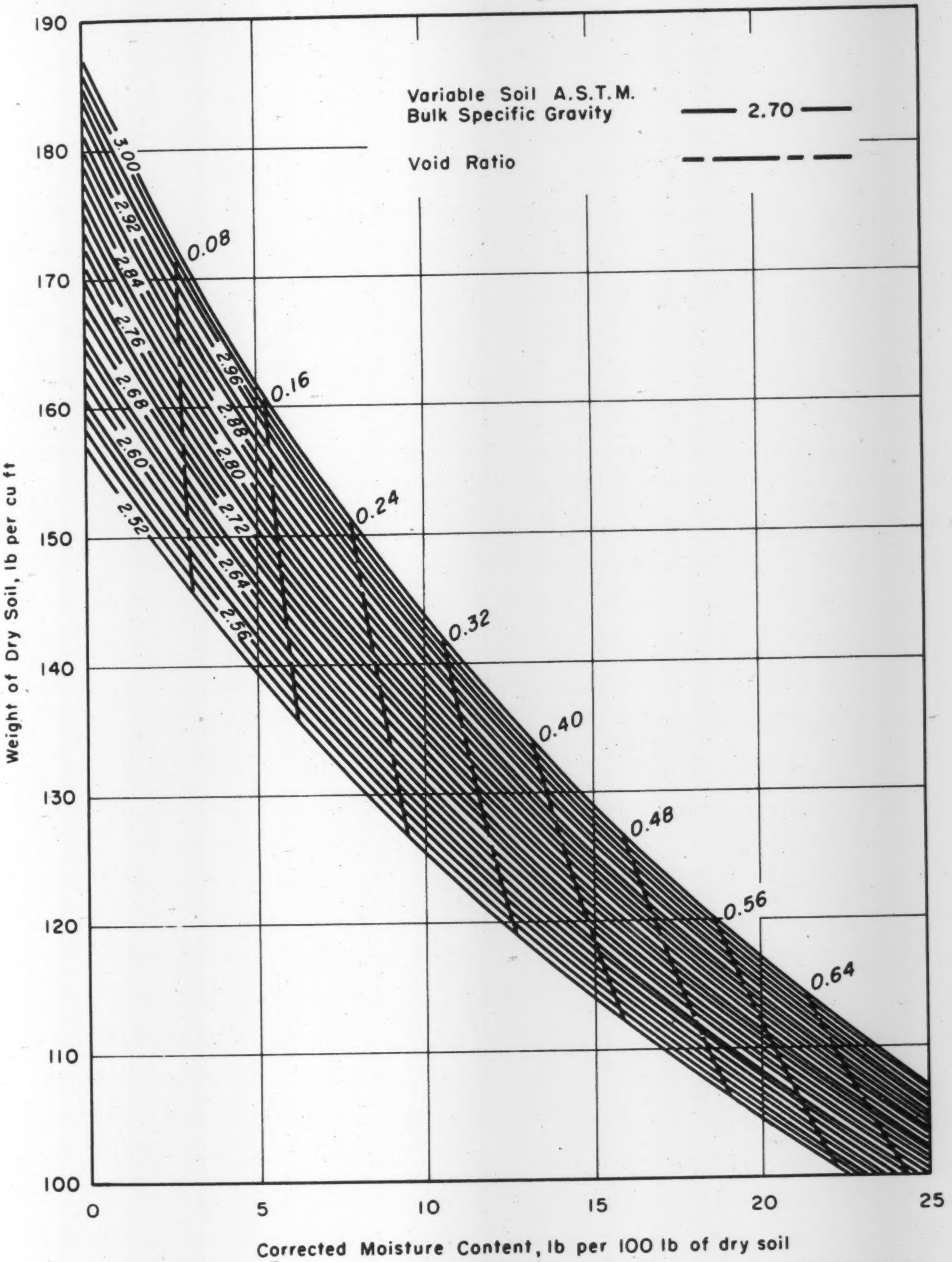

Fig. 14.—Chart for Determining the Void Ratio of a Saturated Soil when the ASTM Bulk Specific Gravity of the Soil is Known.

ways to obtain useful information concerning a saturated soil.

1. In the absence of experimental error, the point representing the oven-dry density and the "corrected moisture content" of the soil must lie on the zero air voids curve corresponding to the ASTM bulk specific gravity of the soil. The location of this point enables the "void ratio" for the soil to be read off by visual interpolation. For example, if the oven-dry density of a saturated soil is 120 lb per cu ft and the corrected moisture content is 16 per cent, these values intersect on the zero air voids curve for a soil with an ASTM bulk specific gravity of 2.78, and the value for the "void ratio" is 0.45.

2. For fine-grained soils, there are no current standard methods for measuring either the amount of water absorbed by the individual soil particles or the ASTM bulk specific gravity of the soil. However, Fig. 14 can be employed to provide the apparent specific gravity of a fine-textured soil. The procedure consists of entering the abscissa of Fig. 14 at the measured moisture content and the ordinate at the dry density of the soil sample. The intersection of these two values enables the apparent specific gravity to be determined by interpolation. For example, suppose the dry density of the saturated soil is 102.5 lb per cu ft and its measured (oven-dry) moisture content is 22.0 per cent. The intersection of these ordinate and abscissa values occur in Fig. 14 on the zero air voids curve for a specific gravity of 2.58. However, when obtained in this way, 2.58 is the apparent specific gravity of the soil, and not the ASTM bulk specific gravity unless there is no water absorption by the individual particles.

When used in this way (total moisture content for the abscissa), the point representing measured moisture and dry density does not provide the correct "void ratio." Some indication of the margin of error in the "void ratio" value that would occur when using Fig. 14 in this manner for fine-textured soils (measured instead of corrected moisture content), when the moisture absorption of the individual soil particles is 2.1 per cent, has been illustrated by Table III, which pertained to a fine-grained soil with a relatively high moisture content.

3. When there is no absorption of water into the individual soil particles, measured and corrected moisture contents for the soil are the same and ASTM bulk and apparent specific gravities are equal. Under these conditions, the dry density and measured moisture content will enable the correct "void ratio" value of the saturated soil to be read directly from Fig. 14.

SUMMARY

1. The advantages of analyzing the composition of soils on a volume rather than on a weight basis are pointed out.

2. Volumetric analysis encourages the calculation of values for void ratio, per cent air voids, and per cent saturation.

3. If the soil particles absorb no water, correct values for these void properties are readily determined.

4. For absorptive soils, examples are given to demonstrate the magnitude of the errors that can occur when evaluating void properties by current methods very often employed for this purpose.

5. Correct values for the void properties of absorptive soils can be obtained only when the volumetric analysis is based on the ASTM bulk specific gravity of the soil, and when full allowance is made for the quantity of water absorbed by the individual soil particles.

6. Charts containing families of curves representing a wide range of values for void ratio, per cent air voids, and per cent saturation, and for which the ordinate is either dry density or corrected wet density, and the abscissa is corrected moisture content, are included.

7. As soon as the point representing the dry or corrected wet density and the corrected moisture content of a soil sample has been plotted on the appropriate chart, correct values for void ratio, per cent air voids, and per cent saturation, can be read directly from the chart by interpolation. This eliminates the time and effort ordinarily required to calculate them.

8. These charts can be usefully employed in connection with seepage, permeability, capillarity, and frost action studies, the design and control of soil compaction, drainage, and filter installations, etc., in addition to their utility for routine soil analysis.

Acknowledgments:

The subject matter of this paper forms part of an extensive investigation of airports in Canada that was begun by the Canadian Department of Transport in 1945. Air Vice-Marshall A. de Niverville, Director of Air Services, has the general administration of this investigation. It comes under the direct administration of Mr. Harold J. Connolly, Chief Construction Engineer, Mr. George W. Smith, Assistant Chief Construction Engineer, and Mr. D. A. Lane. In their respective districts, the investigation is carried on with the generous cooperation of District Airway Engineers G. T. Chillcott, E. B. Wilkins, L. Millidge, R. A. Bradley, F. L. Davis, and W. D. G. Stratton.

Grateful acknowledgment is made to Mr. C. L. Perkins for the able and careful manner in which he prepared the charts for this paper.

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(4) Norman W. McLeod, Discussion on J. H. Dillard's paper "Comparison of Density of Marshall Specimens and Pavement Cores," *Proceedings*, Assn. Asphalt Paving Technology, Vol. 24, p. 210 (1955).

(5) Norman W. McLeod, "Fundamentals of Flexible Pavement Design," *Proceedings*, Second Annual Bituminous Conference, University of Minnesota, Minneapolis, Minn., Nov., 1955.

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DISCUSSION

Mr. W. G. Shockley.[1]—The charts appear to be based on the premise that the voids in the aggregate particles are always filled with water. What evidence is there, particularly at low moisture content in an air-water-soil mixture, that those voids are filled with water?

Mr. N. W. McLeod (*author*).—That is a very good question and is one that might develop some debate. However, because of forces associated with the energy of the heat of wetting and the very high suction pressures of minute capillaries, we believe that, in general, at low moisture contents water will tend to be driven or sucked into the water-permeable porosity of the individual soil particles before it begins to fill the relatively much larger intergranular spaces between the particles. Under equilibrium conditions, the principles of thermodynamics would seem to indicate this to be a reasonable assumption.

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[1] Corps of Engineers, Waterways Experimental Station, Vicksburg, Miss.

MR. SHOCKLEY.—It appears that there should be a lower moisture content limit below which the charts would not apply.

MR. MCLEOD.—The point that Mr. Shockley has mentioned is one that needs to be considered. However, as just pointed out, we believe that at low moisture contents, any available water will move to satisfy the capillary forces of the water-permeable porosity of the individual soil particles first. If this assumption is correct, there is no moisture content below which the charts do not apply. They are applicable to the entire range of soil moisture content.

Figures 5 to 8, 10, and 11 are applicable to any soil with an ASTM bulk specific gravity of 2.65, regardless of its water absorption value. For each of these, the abscissa is "corrected moisture content." To obtain this "corrected" moisture content, the absorbed moisture must be subtracted from the total water content. Similarly, for Figs. 6, 8, and 11, from the measured density, the weight of the absorbed water must be subtracted to determine the "corrected wet density."

The point referred to by Mr. Shockley must receive special consideration when preparing charts like those of Figs. 12 and 13, for which the ordinate is dry or measured wet density, and the abscissa is measured (total) moisture content. Figures 12 and 13 are restricted to soils with a water absorption of 2 per cent (and ASTM bulk specific gravity of 2.65). As indicated at the left-hand side of Figs. 12 and 13, discontinuities occur in the curves for "per cent air voids" and "per cent saturation" at a measured moisture content of 2 per cent, which is the moisture absorption value in this case. However, even for Figs. 12 and 13, there is no lower limit of moisture content below which the charts cannot be applied.

It might be worth while to point out again, that the water-permeable porosity of the individual soil particles, and its content of either air, or water, or both have been excluded when evaluating void ratio, per cent air voids, and per cent saturation. Consequently, the charts have been constructed on the basis that "void ratio" is concerned solely with the intergranular space between soil particles whose volume is calculated from their ASTM bulk specific gravity, "per cent air voids" pertains exclusively to the intergranular air voids, and only the water contained in the intergranular void space influences "per cent saturation."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON IN-PLACE SHEAR TESTING OF FOUNDATION SOIL BY THE VANE METHOD

The so-called vane method for making in-place shear tests of foundation soils has aroused considerable interest in this country. A number of individuals have been making use of this device which was developed in the Scandinavian countries. Apparatus has been imported and in some cases has been devised in this country. The Symposium provided an opportunity for outlining the various considerations involved and for interesting discussion.

Subcommittee R-2 on Sampling and Related Field Testing for Soil Investigation of Committee D-18 on Soils for Engineering Purposes, sponsored the Symposium, held at the thirtieth session of the Fifty-ninth Annual Meeting of the Society in Atlantic City, N. J. on June 22, 1956. J. O. Osterberg, Technological Institute, Northwestern University, Evanston, Ill., acted as Chairman of this Symposium.

The Symposium includes the following papers:

Vane Testing for Shear Properties of Soil—The Present Status of Vane Testing—J. O. Osterberg
An Apparatus and Method of Vane Shear Testing of Soils—Harold J. Gibbs
Deep Vane Tests in Gulf of Mexico—Carl W. Fenske
Vane In-Place Soil Shear Measuring Device—W. A. Hill

The general discussion included three prepared discussions received from Scandinavian sources.

These papers with discussions were issued as *ASTM Special Technical Publication No. 193*, entitled "Symposium on In-Place Shear Testing of Foundation Soil by the Vane Method."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON SPECIFIC GRAVITY OF BITUMINOUS COATED AGGREGATES

Specific gravity has an important role in the present-day design and measurement of compaction of bituminous pavements. It is customarily used in determining compaction of the bituminous mixtures. In the past it has been the practice to measure individually the specific gravity of each component in order to determine the correct mixture proportions. This has not been an accurate or satisfactory procedure.

A symposium comprising six papers was sponsored by Subcommittee B-14 on Specific Gravity of Fine and Coarse Aggregates of Committee D-4 on Road and Paving Materials. The Symposium was held at the first session of the Fifty-ninth Annual Meeting of the Society in Atlantic City, N. J. on June 18, 1956. H. T. Williams, Duquesne Slag Products Co., Youngstown, Ohio acted as Chairman of this Symposium.

The Symposium included the following papers:

- The Role of Specific Gravity in the Design and Control of Bituminous Paving Mixtures—C. A. Carpenter
- Measurement of Bituminous Concrete Core Specific Gravities and Specific Gravities of Bituminous Coated Coarse Aggregates by Means of a Volumeter—F. M. Williams, L. E. Grimmer, M. M. McAdams, and S. Yanoshek
- Measurement of Maximum Theoretical Specific Gravity of a Bituminous by Solvent Immersion—Paul J. Serafin
- Development and Application of the Effective Specific Gravity of Bituminous Coated Aggregates—J. Rogers Martin and Andrew H. Layman, Jr.
- Maximum Specific Gravity of Bituminous Mixtures by Vacuum Saturation Procedure—James M. Rice
- Specific Gravity and Voids Relationships in Bituminous Pavement Mix Design—J. L. McRae

These papers with discussions were issued as *ASTM Special Technical Publication No. 191*, entitled "Symposium on Specific Gravity of Bituminous Coated Aggregates."

THE EFFECT OF STATIC MEAN STRESS ON THE DAMPING PROPERTIES OF MATERIALS*

By Neal L. Person[1] and Benjamin J. Lazan[1]

Synopsis

New bending vibration decay equipment is described for determining the effect of static mean stress on the damping of materials. Tests were performed on SAE 1020 steel, 2024-T4 aluminum, J-1 magnesium, annealed RC 55 titanium, S-816 alloy, glass laminate, and type 403 alloy. The maximum stress on the specimen was kept below the cyclic stress sensitivity limit, below which damping is unchanged by stress history. Type 403 alloy was the only material that displayed a significant change in its damping *versus* alternating stress relationship due to the superimposed static mean stress. Whereas for the other materials the change was less than 30 per cent, for the magneto-mechanical alloy 403 at an alternating stress of 10,000 psi the specific damping energy decreased 90 per cent when the mean stress was increased from 0 to 40,000 psi.

Energy dissipation by means of damping is often one of the most effective methods of minimizing dangerous vibrations under near-resonant operating conditions. In some cases the only type of damping readily available for this purpose is the internal damping of materials. The effect of various test variables on the damping properties of materials is therefore of considerable design significance.

Vibrational stresses resulting from near-resonant operation are often encountered under conditions which impose static mean stresses on the parts. However, very few data are available on the damping properties of materials under various combinations of alternating stress to mean stress. In view of the importance of mean stress in several engineering situations (the blade problems, for example) work was undertaken on the effect of mean stress on damping. Test data reported in this paper considers the case where the maximum stress (mean plus alternating) was kept below the cyclic stress sensitivity limit (**1**)[2] which is approximately 80 per cent of the fatigue limit for many materials.

In prior work the effect of static stress on material damping was investigated using the torsional vibration decay method on wire specimens with the static mean stress applied longitudinally. This method was used by Cochardt (**2, 3**) on several ferromagnetic materials and one nonferromagnetic material. The results of his room temperature tests showed that at lower torsional vibrational stresses the damping in ferromagnetic materials was reduced

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] Research Assistant and Professor of Materials Engineering, respectively, University of Minnesota, Minneapolis, Minn.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1409.

TABLE I.—CHEMICAL COMPOSITION, PRODUCTION, AND TREATMENT.

Name of Material		Chemical Composition, per cent	Production, Heat Treatment
SAE 1020 Steel	Crucible Steel Co., Syracuse, N. Y.	0.3 to 0.6 Manganese, 0.15 to 0.25 Carbon, 0.055 (max) Sulfur, 0.45 (max) Phosphorus, (bal) Iron.	Hot-rolled bar stock $1\frac{5}{8}$ in. diam of electric melted steel, specially controlled for uniformity and cleanliness.
2024-T4 Aluminum	Aluminum Company of America, New Kensington, Pennsylvania	4.30 Copper, 0.22 Iron, 0.14 Silicon, 0.61 Manganese, 1.54 Magnesium, 0.01 Zinc, 0.01 Chromium, 0.01 Titanium, (bal) Aluminum.	Extruded bars $1\frac{1}{4}$ in. diam, solution heat-treated at 920 F, artificially aged for 24 hr at 250 F.
J-1 Magnesium	Dow Chemical Co., Midland, Mich.	5.8 to 7.2 Aluminum, 0.15 (min) Manganese, 0.4 to 1.5 Zinc, 0.3 (max) Silicon, 0.05 (max) Copper, 0.005 (max) Nickel 0.005 (max) Iron, 0.3 (max) other, (bal) Magnesium.	Extruded bar $1\frac{5}{8}$ in. diam, used in this condition.
RC 55 Titanium (annealed)	Rem-Cru Titanium Inc., Midland, Pa.	0.045 Carbon, (bal) Titanium.	Initially forged in temperature range 1700 to 1800 F, hot rolled to $\frac{7}{8}$ in. diam. in temperature range 1450 to 1550 F and finally annealed for 1 hr at 1300 F.
S-816 Alloy	Allegheny Ludlum Steel Corp., Pittsburgh, Pa.	0.397 Carbon, 1.12 Manganese, 0.50 Silicon, 19.62 Chromium, 20.62 Nickel, 4.10 Molybdenum, 0.018 Sulfur, 0.012 Phosphorus, 2.86 Columbium, 4.03 Wolfram, 2.99 Iron, 42.9 Cobalt, 1.03 Tantalum.	$\frac{3}{8}$ in. annealed and ground, 1 hr at 2300 F, quenched in water, aged at 1400 F for 16 hr, furnace cooled.
Type 403 GE B50R 207 Alloy	Republic Steel[a] Corp., Cleveland, Ohio	0.12 Carbon, 0.50 Manganese, 0.12 Phosphorus, 0.16 Sulfur, 0.35 Silicon, 12.12 Chromium, 0.25 Nickel, 0.04 Molybdenum, 0.009 Aluminum, 0.008 Stannum, (bal) Iron.	Electric furnace alloy hot-rolled to 1 in. round. Heated to 1750 F (maintained for 15 min), quenched in oil. Tempered at 1050 F for $1\frac{1}{2}$ hr. Air cooled.
Glass Laminate Plastic	Cincinnati Testing[b] and Research Laboratory, Cincinnati, Ohio	CTL-91-LD Resin, 181–114 Fiberglass.	Resin impregnated into fiberglass fabric and pressed at approximately 15 psi into panel $1\frac{1}{4}$ in. thick.

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[a] Procured through General Electric Co., Metallurgical Development Unit, Plant Laboratory, Building 200, Lockland 15, Ohio.

[b] Procured through Wright Air Development Center, Wright-Patterson Air Force Base, Ohio, Attention: WCRTL-5, Materials Laboratory.

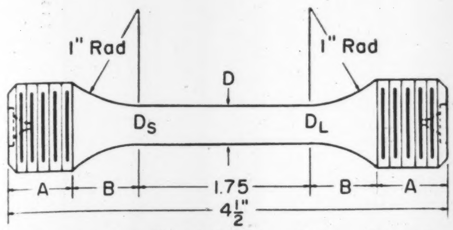

FIG. 1.—Bending Vibration Decay Damping Specimens.

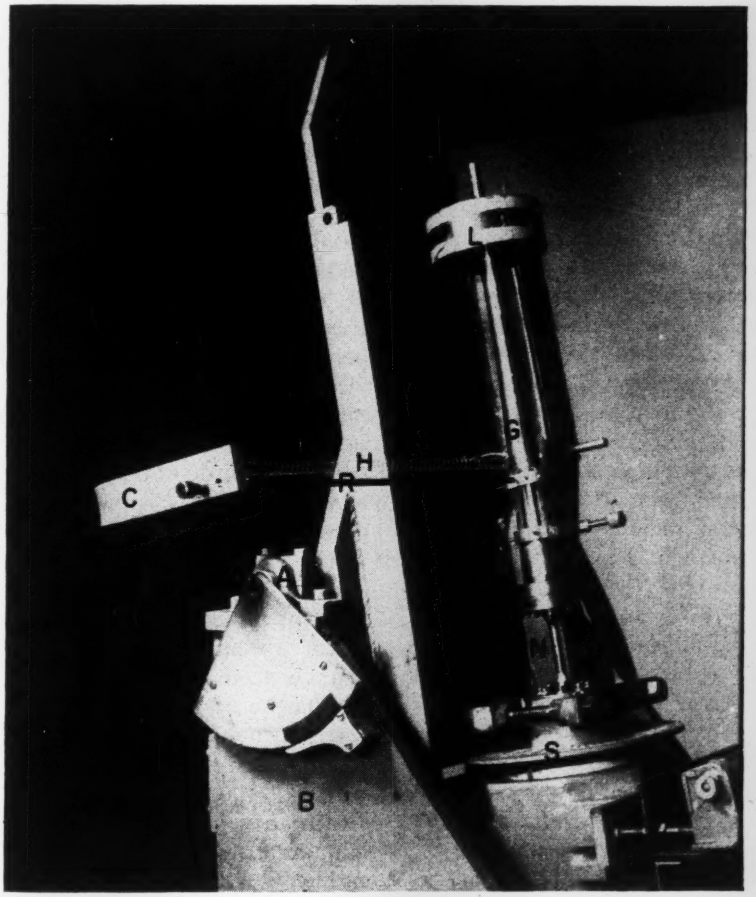

FIG. 2.—Rotating Cantilever Beam Fatigue and Damping Testing Machine Adapted for Static Mean Stress Vibration Decay Tests.

with increased tensile static stress, whereas the damping increased with static stress for the non-ferromagnetic material. It should be emphasized however that in this type of test the principal stress planes for the static preload associated with the tension are displaced 45 deg from the principal stress planes for the alternating stress associated with the torsion. It was therefore believed desirable to procure data on the effect of mean stress under conditions in which the principal stress planes are the same for both the static prestress and the alternating stress. This is the condition present in most engineering problems, such as turbine blades for example.

The test program consisted of running bending vibration decay tests on each of the materials at various values of static mean bending stress. All tests were conducted at room temperature. The maximum total stress at the beginning of each test was kept below the cyclic stress sensitivity limit in order to eliminate the effect of stress history. Zero mean stress tests were run before and after the other mean stress tests to check the effect of stress history.

TEST MATERIALS AND SPECIMENS

The materials tested in this program were SAE 1020 steel, 2024-T4 aluminum, J-1 magnesium, annealed RC 55 titanium, S-816 alloy, glass laminate, and type 403 alloy. This particular group was selected to give a variation in material type and, therefore, in damping properties. Details on the chemical composition, processing, and heat treatment of the materials are given in Table I.

The test sections of the specimens were tapered ($D_L > D_S$) so as to produce a constant maximum stress along the outer surface under the cantilever loading (1). Specimen dimensions are given in Fig. 1, in which average diameter D was 0.585 in. for the magnesium and aluminum, 0.375 in. for the mild steel, and 0.250 in. for the remaining materials.

The test sections of the specimens were prepared using the following general procedure. First the specimen stock was turned to 0.002 to 0.005 in. oversize. Next, the section was rough polished to 0.0005 in. oversize using a 240 grit belt and finally was finish polished to size using a 400 grit belt. Details of this method of polishing have been published previously (4).

VIBRATION DECAY EQUIPMENT AND TEST PROCEDURE

A rotating cantilever machine described in previous publications (1, 4), normally suitable for reversed stress testing only, was adapted for mean stress work by converting it for bending vibration decay tests as follows.

The rotating-beam testing machine was mounted on a 4000-lb concrete block suspended on helical springs in order to isolate the vibrating system from foreign vibrations and energy. The upper part of the machine is shown in Figs. 2 and 3. Spindle S, table P, and frame B were all clamped firmly together to minimize energy loss in the specimen supporting system. The desired bending mean stress was imposed on the specimen by rotating table P about axle A to a predetermined angle with the vertical so that the arm G-weight L combination acquired the necessary moment arm about the center of the specimen. If the weight-arm-specimen combination is now displaced perpendicular to the axle and allowed to vibrate freely, the specimen will feel a mean bending stress dependent on the table angle and a cyclic stress dependent on the amplitude of vibration. The rate of vibration decay is proportional to the damping energy absorbed by the specimen.

One of the difficulties encountered was that the direction of vibration for cylindrical specimens had a tendency to change from the displacement plane during the decay of motion. In order to restrain the vibrations to a single plane, a light rod R with sharp conical ends and a soft tension spring H were placed between the vibrating arm G and a rigid support arm C. The spring maintained rod contact and thus confined the vibrations to a vertical plane perpendicular to axle A.

The displacement of the vibrating system was measured with a linear variable differential transformer. The transformer core was mounted on the vibrating arm and the coil was fixed to the table. The relation between the arm displacement and the recorder displacement was found to be very nearly linear, even though the core moved through a small arc. The output of this transducer was recorded with a strain gage amplifier unit having adequate frequency response exceeding 40 cps. Since none of the decay test frequencies used were higher than 6 cps, the decay curves obtained were reliable.

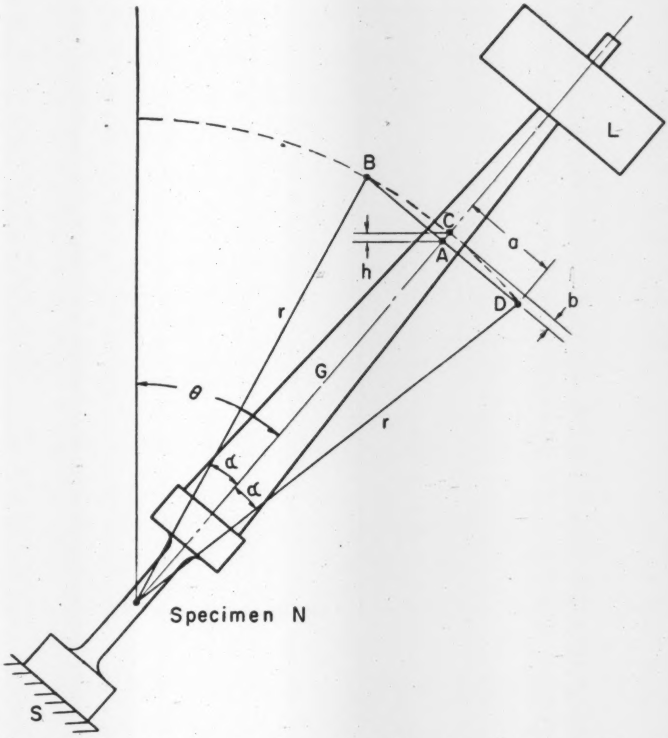

Fig. 3.—Schematic Diagram of Vibration Decay Arm, Weight, and Specimen.

The location of resultant inertia force produced by the vibrating arm-weight system, that is its center of percussion, was considered in the design of specimen taper. Thus the maximum stress along the test section of the specimen was essentially constant.

Air losses and the dissipation of energy in the supporting system are sometimes serious sources of error in flexural vibration decay tests (7). The investigation of these and other sources of energy loss is discussed in Appendix II, which indicates these errors are relatively small under the test conditions employed.

As a further check on the reliability of the vibration decay method, comparison tests were performed on rotating-beam equipment. The agreement between these two types of tests, on the basis of *specific* damping energy (see section on reduction of data) was considered adequate (15).

The following general test procedure was used on all seven test materials. The specimen was statically deflected to a position which produced a maximum total stress approximately 10 per cent below the cyclic stress sensitivity limit, a thin wire being used to maintain this deflection. The tests were started by releasing the arm from this deflected position by cutting the wire with an acetylene torch. This method of release did not excite the supporting system or produce harmonic or other stray disturbances.

Tests at zero mean stress were run periodically before and after the other mean stress tests to detect the presence of any stress history effects. Also, several tests were run at each mean stress to check the reproducibility of results.

REDUCTION OF DATA

The logarithmic decrement δ_{og} may be computed from the test data using the following simplified equation:

$$\delta_{og} = \frac{2}{N}\frac{A_1 - A_N}{A_1 + A_N} \quad \text{.........(1)}$$

A definition of the symbols is given in Appendix I. When δ_{og} is less than 0.05, which is usually the case, the error involved in this simplified equation is less than 2 per cent.

A correction for work done by the vibrating system against gravity is required for the vibration decay tests under mean stress. The origin of this energy loss and its determination is indicated in Fig. 3. As the system vibrates about mean stress angle θ, its center of gravity, point C, moves along the path BCD. Point A may be defined as the mean static position of the center of gravity during vibration between points B and D. It is observed that as the amplitude of vibration decreases, the mean position A approaches C against the force of gravity. The energy associated with this elevation of point A is subtracted from the energy in the vibrating system. This work done against gravity produces a decay effect similar to that caused by system damping.

This work per cycle D_g done against gravity is equal to the change in distance h per cycle (see Fig. 3) multiplied by the weight of the system, W. It can be shown (15) that:

$$h = r \cos\theta\,(1 - \cos\alpha)$$

and the change in h per cycle is:

$$\Delta h = h_1 - h_2 = r\cos\theta\,(\cos\alpha_2 - \cos\alpha_1) \quad \text{..(2)}$$

Since the angle α is small, $\alpha \cong a/r$ and:

$$\Delta h \cong \cos\theta\left(\frac{a_1^2 - a_2^2}{2r}\right) \quad \text{.......(3)}$$

and the work per cycle against gravity is:

$$D_g = \frac{W\cos\theta}{2r}\,(a_1^2 - a_2^2) \quad \text{.......(4)}$$

It is observed that at $\theta = 0$ the work is a maximum and at $\theta = 90$ deg no work is done. For θ between 90 deg and 180 deg, the correction is negative and gravitational energy is added to the vibrating system.

The logarithmic decrement calculated from the decay curve must be corrected for the gravity effect. The decrement δ_{og} observed during a test is related to the system energies by:

$$\delta_{og} = \frac{D_{og}}{2W_o} \quad \text{.............(5)}$$

By contrast, the logarithmic decrement δ associated with the specimen damping only is:

$$\delta = \frac{D_o}{2W_o} \quad \text{.............(6)}$$

Since $D_o = D_{og} - D_g$, the true logarithmic decrement associated with specimen damping is:

$$\delta = \frac{D_{og} - D_g}{2W_o} = \delta_{og} - \frac{D_g}{2W_o} \quad \text{.....(7)}$$

This is the logarithmic decrement used in the equations which follow.

The logarithmic decrement δ as defined above may be converted to specific damping energy, considered to be the fundamental unit for expressing damping (16), as follows:

The total damping D_o in the specimen may be expressed as:

$$D_o = \int_0^{V_0} D\, dV$$

$$= \frac{V_o}{S_o} \int_0^{S_0} D \frac{d(V/V_o)}{d(S/S_o)}\, dS \quad \ldots\ldots\ldots\ldots (8)$$

Assuming that (*a*) the stress distribution along longitudinal direction is uniform and (*b*) the stress-strain relationship on cross section is linear, then:

$$\frac{V}{V_o} = \frac{2}{\pi}\left[\frac{S}{S_o}\sqrt{1-\left(\frac{S}{S_o}\right)^2} + \sin^{-1}\frac{S}{S_o}\right]$$

$$\frac{d(V/V_o)}{d(S/S_o)} = \frac{4}{\pi}\sqrt{1-\left(\frac{S}{S_o}\right)^2}$$

Thus Eq 8 may be written as:

$$D_o = \frac{4}{\pi}\frac{V_o}{S_o}\int_0^{S_0} D\sqrt{1-\left(\frac{S}{S_o}\right)^2}\, dS \quad ..(9)$$

In order to obtain a closed solution for Eq 9, a function for the specific damping energy in terms of stress must be assumed. In the stress region below the cyclic stress sensitivity limit, the damping of most materials may be expressed as follows (16):

$$D = JS^n \quad \ldots\ldots\ldots\ldots (10)$$

Under these conditions it can be shown that:

$$D_o = \frac{4}{\pi}\frac{V_o}{n+2} D \int_0^{\pi/2} \sin^n \psi\, d\psi$$

where ψ is the arc sine of S/S_o and since $n > -1$, the specific damping energy is:

$$D = \frac{D_o}{V_o}\frac{\sqrt{\pi}(n+2)}{2}\frac{\Gamma\left(\frac{n+2}{2}\right)}{\Gamma\left(\frac{n+1}{2}\right)} \quad ..(11)$$

But for range of small values of δ considered here

$$D_o = 2\delta W_o = \delta PX$$

or

$$D = \frac{\delta PX}{V_o}\frac{\sqrt{\pi}(n+2)}{2}\frac{\Gamma\left(\frac{n+2}{2}\right)}{\Gamma\left(\frac{n+1}{2}\right)} \quad ..(12)$$

Equation 12 indicates that the actual reduction of data may be simplified in some cases. If the plot of log δ *versus* log amplitude A (or stress S_o) is a straight line with a slope n', the value of n in Eq 10 is a constant and is equal to $n' + 2$ (both P and X vary as the first power of stress). The log D *versus* log S_o curve may then be obtained by calculating D at any given stress by use of Eq 12 and then drawing a straight line of slope n through this point.

In some cases (403 alloy, for example) the log plot of δ and A is not a straight line (n not constant) below the cyclic stress sensitivity limit. For these cases the specific damping energy was calculated at various stresses using a local value of n in Eq 12. A smooth curve was then drawn through the calculated points. Even though this approach is an approximation the error is not considered to be large for the range on n's encountered to date below the cyclic stress sensitivity limit (17).

It would be misleading to show the plotted points on the specific damping energy *versus* alternating stress curves because all the points would fall on the curves. This is due to the fact that the values of the logarithmic decrement used in Eq 12 are taken from a smooth curve drawn through the δ *versus* stress plot, which does show scatter of data. The range of this scatter and thus the range of scatter of the damping curves is ±7 per cent or less for all the materials tested.

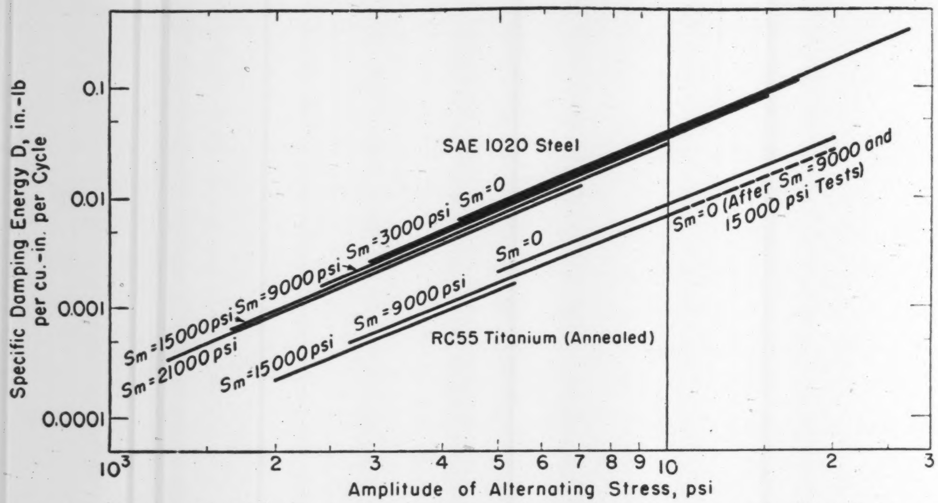

FIG. 4.—Effect of Static Mean Stress, S_m, on Specific Damping Energy for SAE 1020 Steel and RC55 Titanium (Annealed).

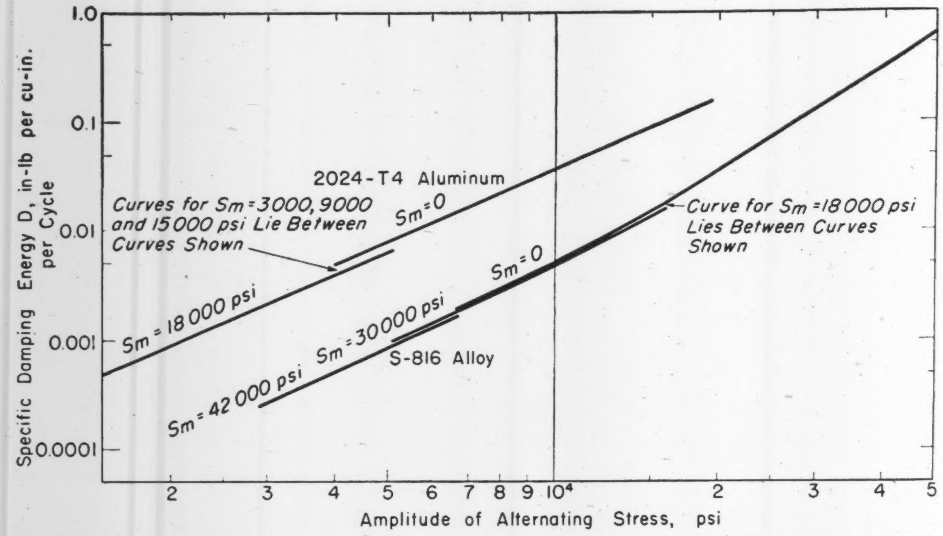

FIG. 5.—Effect of Static Mean Stress, S_m, on Specific Damping Energy for 2024-T4 Aluminum and S-816 Alloy.

TEST RESULTS AND DISCUSSION

The effect of mean stress on damping is shown by a series of plots, Figs. 4 to 7, which show the log-log relationship between specific damping energy D and alternating stress for each of several mean stress levels. If the curves for various stress levels fall together, this indicates that damping is primarily dependent on alternating stress only and is affected very little by mean stress. Separation of the lines indicates a mean stress dependence.

Figure 4 shows the results of the mean

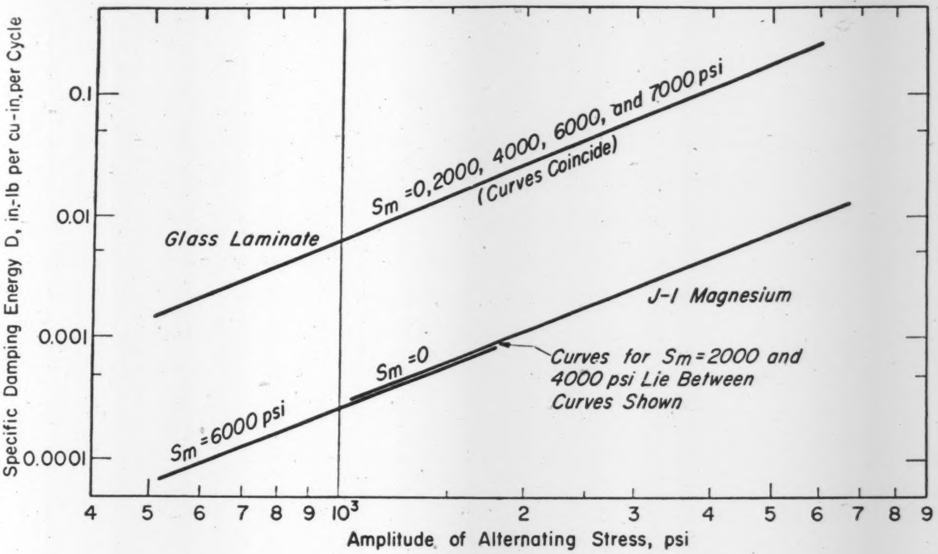

FIG. 6.—Effect of Static Mean Stress, S_m, on Specific Damping Energy for Glass Laminate and J-1 Magnesium.

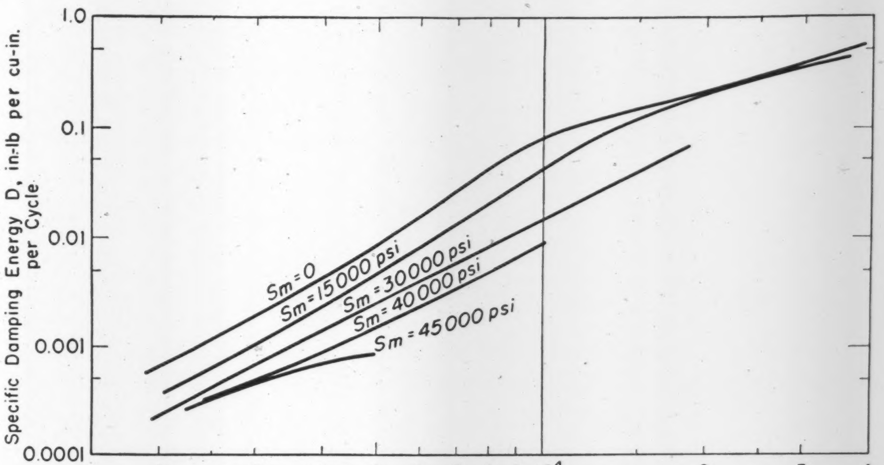

FIG. 7.—Effect of Static Mean Stress, S_m, on Specific Damping Energy for Type 403 Alloy.

stress tests on 1020 steel. It is observed that the damping energy corresponding to any given alternating stress decreases very little with increased mean stress. At the maximum mean stress tested, 21,000 psi, a decrease of only 30 per cent below the zero mean stress value is observed. This is an extremely small difference compared with the effect of alternating stress; for example, the damping at 2000 psi is only 1 per cent of the value at 23,000 psi (a 21,000 psi difference).

The other test materials in which

ferromagnetic damping effects are small show similar behavior, changing very little with increased prestress as shown in Figs. 4, 5, and 6. The maximum change in damping for all these materials is 30 per cent.

As mentioned previously, the maximum stress in all tests was kept below the cyclic stress sensitivity limit; thus, no change in damping with stress history was expected, nevertheless, RC 55 titanium did undergo a small change in damping due to stress history. Figure 4 shows the curve for zero mean stress (coincides with the 9000 psi mean stress curve) obtained after all other tests had been completed. The difference associated with stress history is thus less than 20 per cent.

The tests on glass laminate indicate that the same damping-stress curve is obtained whether the laminations are parallel or perpendicular to the plane of vibration.

The ferromagnetic material 403 alloy shows a very significant decrease in damping with increased preload. Figure 7, which gives the test results for 403 alloy, indicates that for the range of alternating stresses covered *D* decreased about 90 per cent when the mean stress was increased from zero to 40,000 psi.

The fact that type 403 alloy displayed a definite decrease in damping with increased static mean stress agrees with the work by Cochardt (2, 3) and may be explained by the domain theory of ferromagnetism (8). The effect of a static stress on the damping of a ferromagnetic material is similar to that of a constant magnetic field (9, 10).

The results of the tests on the five materials without large ferromagnetic effect differ from the results obtained by Cochardt (3) who observed that significant increase in damping occurred with static stress for Refractaloy 26. This disagreement is believed to be caused by the difference between the two testing methods. The method used by Cochardt involved a static tensile stress with superimposed alternating torsional stress, whereas, the method used in this program imposed static bending stress and alternating bending stress on the same planes. In the Cochardt tests the principal stress planes for the static stress were displaced 45 deg from the principal stress planes for the alternating stress, while in the case of combined static and alternating bending stresses, the principal stress planes for the two types of stress coincide.

CONCLUSIONS

The data presented lead to the following conclusions for the materials investigated in the range of maximum stress (mean plus alternating) up to the cyclic stress sensitivity limit.

1. The effect of a static mean bending stress on the damping associated with a given alternating bending stress is small for materials with little magneto-mechanical effect. A maximum decrease in damping of only 30 per cent was observed for nonferromagnetic materials.
2. Increased static mean stress greatly reduces the damping of type 403 alloy which has considerable magneto-mechanical damping. The specific damping energy was decreased as much as 90 per cent with increasing static mean stress.

It should be emphasized that this work was conducted below the cyclic stress sensitivity limit, which is approximately 80 per cent of the fatigue strength, and at room temperature.

Acknowledgments:

This work was sponsored by Wright Air Development Center, and the fine cooperation of W. J. Trapp of Wright Air Development Center is greatly appreciated.

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APPENDIX I

DEFINITION OF SYMBOLS AND TERMS

A_N = amplitude of N$^{\text{th}}$ cycle of vibration, in.

$a, b, h, r, \alpha, \theta$ = distances and angles shown in Fig. 3, in. and deg.

D = specific damping energy of a material or that associated with a specific stress, in.-lb per cu in. per cycle.

D_g = energy dissipated during decay test as work against gravity, in.-lb per cycle.

D_o = total damping energy absorbed by specimen per cycle, in.-lb per cycle.

D_{og} = $D_g + D_o$, in.-lb per cycle.

J = Constant in damping stress equation; the damping energy at a stress of 1 psi.

n = slope of log D *versus* log S plot $= n' + 2$.

n' = slope of log δ *versus* log amplitude plot.

N = number of cycles of vibration.

P = normal force acting through the center of percussion necessary to produce a stress equal to the stress associated with the computed value of δ, in lb.

S = unit normal fiber stress at any point in specimen, psi.

S_o = maximum unit normal stress at outside fibers of test section of specimen, psi.

S_m = static mean stress, psi.

V = volume having stress less than S, in cu in.

V_o = total effective volume of specimen contributing to dissipation of energy D_o in cu in.

W = total weight of vibration decay arm, weight and half of specimen.

W_o = total elastic energy in specimen at maximum stress, in.-lb.

X = distance through which force P acts in in.

δ = logarithmic decrement, defined as the logarithm of the ratio of successive amplitudes of decay vibrations

$$\cong \frac{2}{N}\frac{A_1 - A_N}{A_1 + A_N}$$

δ_{og} = logarithmic decrement computed from recorded decay curve.

APPENDIX II

SOURCES OF EXTRANEOUS ENERGY DISSIPATION DURING THE BENDING VIBRATION DECAY TEST

One difficulty in the bending-vibration-decay method of determining material damping is the elimination of extraneous energy losses. Various tests were made in the machine described above to determine the approximate magnitude of the various sources of energy radiation considered to be serious causes of error.

The three most important energy losses studied were: (*a*) air churning, (*b*) support system friction, impact, etc, and (*c*) guide rod and spring friction. Each of these three is discussed below.

The amount of energy dissipated due to air damping depends on the size and shape of the vibrating arm-weight combination, test frequency, and amplitude of vibration. Since air damping varies approximately as the square of velocity it also varies approximately as the squares of test frequency and amplitude.

The effect of air was studied in two manners. First, the damping was increased by attaching a sail-like plate of negligible weight to the arm; and secondly, the damping was decreased by running tests at reduced air pressures. An estimate of the air damping obtained from these studies is given in Fig. 8, which also shows the total damping energy curves for each of the test materials included in the mean stress investigation. Since various types of specimens and weights were used, the total damping energy is plotted against the product of amplitude of vibration, frequency, and frontal area of the moving parts to provide a common abscissa. The air damping error at a given point on any of the

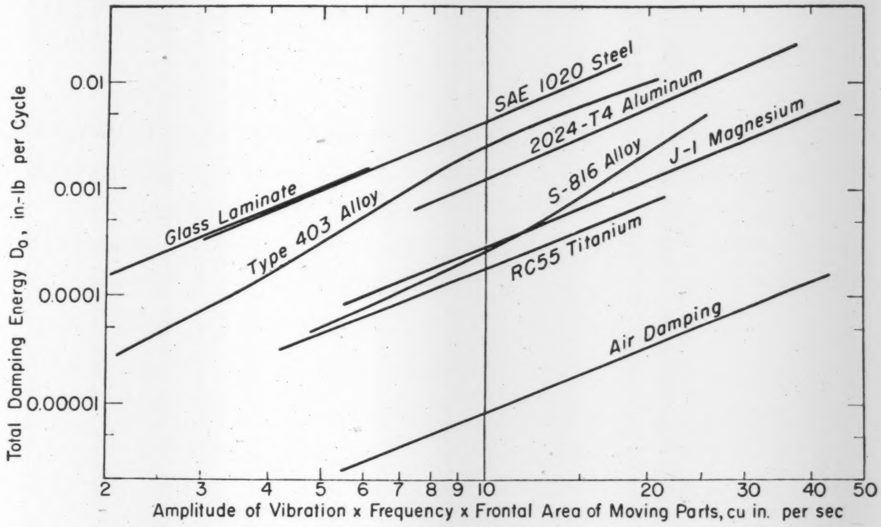

FIG. 8.—Comparison of Material Total Damping Energy and Air Damping Energy.

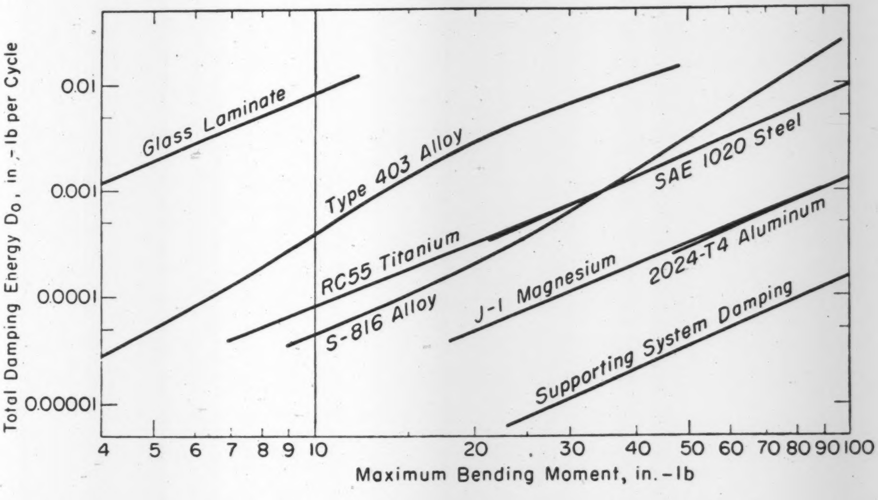

FIG. 9.—Comparison of Material Total Damping Energy and Supporting System Damping Energy.

material damping curves is approximately equal to the value of the air damping curve at the same value of the abscissa. It is evident from Fig. 8 that air damping contributes negligible error to tests. For example, at an abscissa value of 10 in.^{3} per sec, the total damping energy for titanium, which has the lowest total damping curve of the seven materials tested, is 1.90×10^{-4} in.-lb per cycle while the air damping is only 8.5×10^{-6}. This represents an error of only about 4 per cent.

The support system includes the rotating-beam machine, concrete block, and suspension springs. If the block is not at resonance during the tests, no significant energy loss occurs in the supporting springs. However, if the decay test frequency is near one of the natural frequencies of the suspended block, a continuous beat transfer of motion takes place between the vibrating arm and the block; thus the energy loss in the springs may be quite large. These critical frequencies can usually be avoided for any given material by using an appropriate combination of weight size and specimen type.

The exact location of the source of energy dissipation within the rotating-beam machine is unknown. Most likely the energy loss occurs in the specimen grips and the spindle and table bearings. The grip loss is expected to depend on the tightness of the grips, but loosening or tightening them within a range of reasonable values caused no noticeable change in the observed damping; thus this loss appears insignificant. The energy dissipation in the rotating-beam machine, excluding the grips, was investigated by comparing the results of decay tests run in the rotating-beam machine with the test results obtained with the same specimen fixed directly to the concrete block. The energy absorbed by the concrete is insignificant because the stresses produced in the concrete are extremely small. The approximate damping in the supporting system found by this work is shown in Fig. 9. Also shown are the total damping energy curves for the seven test materials mentioned above. In this case total damping energy is plotted against maximum bending moment since the machine energy loss is chiefly dependent on the moment. Figure 9 shows that the J-1 magnesium and 2024-T4 aluminum damping curves are lowest for a given moment and therefore have the greatest error due to the support losses. At a moment of 80 lb-in. the total damping energy for magnesium and aluminum is 8.0×10^{-4} in.-lb per cycle, whereas the supporting system damping is 8.0×10^{-5} in.-lb per cycle, representing an error of 10 per cent.

Friction at the ends of the guide rod causes an energy loss that is dependent primarily on the amplitude of vibration and to some extent on the inertia forces in the system. Tests were run with and without the rod and spring using round specimens with flats machined on opposite sides to assure vibration in a single plane when the guide rod was not used. SAE 4340 steel and S-816 alloy were tested in this manner. A liberal estimate of the guide rod damping derived from this work is shown in Fig. 10 along with the total damping energy curves for the seven materials tested in the mean stress investigation. In this figure damping energy is plotted against amplitude of vibration. For the normal range of amplitude encountered, the rod damping varies from 5.0×10^{-6} to 3.0×10^{-5} in.-lb per cycle, whereas for RC 55 titanium, which has the lowest total damping energy associated with a given amplitude, the total damping energy varies from 3.0×10^{-5} to 6.0×10^{-4} in.-lb per

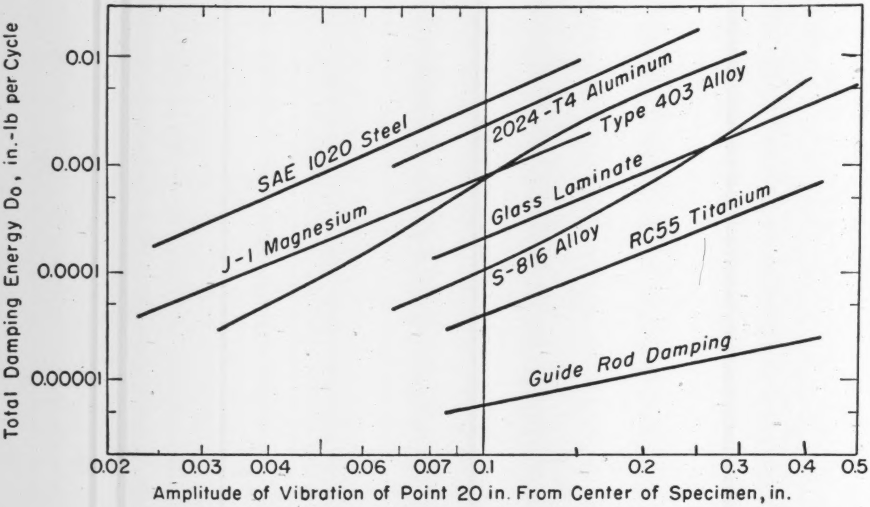

FIG. 10.—Comparison of Material Total Damping Energy and Guide Rod Damping Energy.

cycle. Thus, guide rod friction causes a maximum error of less than 10 per cent in the range of testing for which data are presented.

It is observed from Figs. 8, 9, and 10 that the error percentages given above are maximum values which correspond to only certain materials. For example, the supporting system losses were estimated to be 10 per cent for aluminum and magnesium but for the other materials they amount to less than 3 per cent, see Fig. 9. Therefore, since the three error percentages given above do not all pertain to any one material, the sum of these percentages does not give the true total error. The largest total error is approximately 15 per cent which exists in the tests on RC 55 titanium (4 per cent air damping, 1 per cent support damping, and 10 per cent rod and spring damping.

DISCUSSION

MR. K. WINTERTON[1] (*presented in written form*).—The results of this investigation are interesting because for some years work has been going on at the Ontario Research Foundation aimed at clarifying the relationship between magnetic properties and mechanical behavior and more recently to the utilization of the data for practical benefit.

The authors have shown that with strongly ferromagnetic material, specific damping decreases with increasing mean stress for a given amplitude of alternating stress. This is due to the reduction in magnetomechanical damping. Domain wall movement is inhibited by a static stress as it is also by a magnetic field. In our own tests, techniques have been developed for studying changes in ultrasonic damping and also magnetic damping. A reduction in damping with increasing stress is observed in both instances.

Mention has been made in the paper of the similar action of stress and of a magnetic field. It is as well to remember two important differences. First, while there is no theoretical upper limit to the field strength that can be applied to a specimen, a practical upper limit of stress is imposed from mechanical considerations. Second, the directional effect of a magnetic field is absent in the case of an applied stress, and no magnetization results.

The authors have noted a most pronounced effect for the ferromagnetic stainless steel type 403. In one sense however the effect is not much greater than that observed for the 1020 mild steel. Comparing Figs. 4 and 7 of the paper and examining the changes in damping as the mean stress is raised from 0 to 15,000 psi, the effects are roughly comparable with the two materials. It is only the fact that much higher stresses are permissible with the stainless steel that produces a greater absolute effect. Incidentally, it would be interesting to know the authors' explanation for the curvature of the line in Fig. 7 for zero mean stress at high amplitude. Does this mean that there is a limit to the magnetomechanical damping at high applied stresses?

Theoretical considerations have led us to believe that materials with a large magnetic remanence should be capable of a greater dissipation of magnetic energy and consequently greater magnetomechanical damping. It would be interesting to study the changes occurring in remanence (or more completely in magnetization) under vibrational and fatigue conditions. Perhaps the authors could supply some data relating to the magnetic properties of the material used in their tests.

Magnetomechanical damping supplies an important means for the dissipation of fatigue energy. New alloys can be designed to make the best use of this property with increased resistance to fatigue. For example we conceive an alloy with randomly distributed inclusions which are also single particle magnetic domains. Cobalt could be satisfactory in an appropriate matrix. Such a structure should have good fatigue properties.

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[1] Assistant Director, Department of Engineering and Metallurgy, Ontario Research Foundation, Toronto, Canada.

MR. B. J. LAZAN (*author's closure*).—The authors wish to express their thanks to Mr. Winterton for his comments and observation that tests conducted at the Ontario Research Foundation have also shown that damping decreases with increased stress.

Mr. Winterton states that the effects of increasing the static mean stress from 0 to 15,000 psi for 1020 steel and type 403 alloy are "roughly comparable." Actually the damping of the mild steel decreased about 20 per cent and type 403 alloy decreased about 45 per cent for a mean stress of 15,000 psi. However, if comparisons are made on the basis of range of mean stress within permissible limits, rather than absolute mean stress, a very much larger difference is observed for type 403 alloy. Since this later basis is more closely associated with the range of stresses usually encountered in engineering practice, it is believed to be more meaningful.

In regard to Mr. Winterton's inquiry concerning the curvature of the zero mean stress line for type 403 alloy, a very similar curve has been obtained for this material using a rotating-cantilever-beam machine.[2] It is believed that an upper limit to magneto-mechanical damping does exist which is caused by the alignment of all the domains at some critical stress. This and other aspects of the magneto-mechanical mechanism is discussed in reference (**9**) of the authors' bibliography.

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[2] E. R. Podnieks and B. J. Lazan, "Damping, Elasticity, and Fatigue Properties of Titanium Alloys, High Temperature Alloys, Stainless Steels, and Glass Laminate at Room and Elevated Temperatures," Wright Air Development Center Technical Report, *WADC TR 56-37*.

AN ULTRASONIC SCANNING AND RECORDING SYSTEM*

By W. J. McGonnagle[1] and W. N. Beck[1]

Synopsis

Ultrasonic techniques have been used as a nondestructive test to differentiate between areas of intimate metal-to-metal contact with no bond and areas where two metals are fused together in a metallurgical bond. A test program was undertaken at Argonne National Laboratory to test nondestructively a large number of laminated metallic specimens. Since some of the specimens to be tested are less than 0.100 in. in thickness, it is necessary to use a two-crystal through-transmission technique. The purpose of this paper is to describe a scanning and recording system developed to test for defects and lack of bonding in plate and cylindrical type specimens, using the ultrasonic transmission technique.

The ultrasonic transmission technique requires a separate sending and receiving transducer. A pulsed ultrasonic wave or modulated continuous wave is sent through the specimen positioned between the two transducers and the amplitude of the transmitted wave measured. In general metals readily transmit ultrasonic waves. If the metals are homogeneous, the beam is transmitted without loss of energy due to scattering or reflection. If there are voids, cracks, lack of bonding or other inhomogeneities, a measureable reflection and scattering of the beam takes place at these irregularities. Any reflection and scattering reduces the amount of energy reaching the receiving transducer.

An ultrasonic beam is propagated through a metal in essentially a straight line. This characteristic of ultrasonic waves is due to their short wavelength. As the wavelength becomes shorter and shorter the waves approach more closely the ideal conditions of absolute rectilinear propagation. It is this property of straight line propagation which makes it possible to use ultrasonics for locating defects since any inhomogeneity will cast a rather sharp shadow.

Ultrasonic waves, in common with other forms of radiation, have the property of being reflected effectively by objects having dimensions less than the wavelength of radiation. The higher the frequency the smaller the defect that can be located. However, in general, as the frequency is increased the penetration of the beam is decreased and consequently a practical limit is set to the frequency that can be used successfully. The decreased penetration is due to the scattering of the waves by the grains of the metal. This effect is particularly large when the wavelength of the wave approaches the size of the grains in the metal.

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17-22-1956.

[1] Associate Physicist and Assistant Physicist, respectively, Metallurgy Div., Argonne National Laboratory, Lemont, Ill.

TESTING APPARATUS

The apparatus developed for this testing work consists of an ultrasonic generator and receiver, a scanning assembly, and a recording system.

Generator and Receiver.—The ultrasonic generator and receiver used in this work was a commercial Sperry Reflectoscope. The instrument was modified so that (1) a separate sending and receiving transducer could be used; (2) the pulse repetition rate could be varied; and (3) a negative pulse could be taken from the pulse generator for keying the recording system.

Scanning Assembly.—The scanner assembly consists of a rigidly supported track, a tank, transducer carriage, three drive motors, and two Selsyn motors. Figure 1 shows the scanner with the sheet metal side panels removed.

The track consists of two rails: an inverted V shaped rail and a flat rail. The rails are $10\frac{1}{2}$ ft long and are 20 in. apart. The two rails are accurately aligned and rigidly supported on a welded steel framework.

The tank is 10 ft long, 12 in. high, and 14 in. wide and is made of No. 16 gage steel, spray coated with white Koroseal. This coating serves a two-fold purpose of protecting the tank from corrosion by the couplant and acts as a sound absorbing medium. Tap water is used as a couplant. After the water has been in the tank a few hours, no difficulty has been experienced with gases entrapped in the water.

The transducer carriage is used to support and carry the two quartz crystal transducers along the sample being inspected. The transducer carriage is

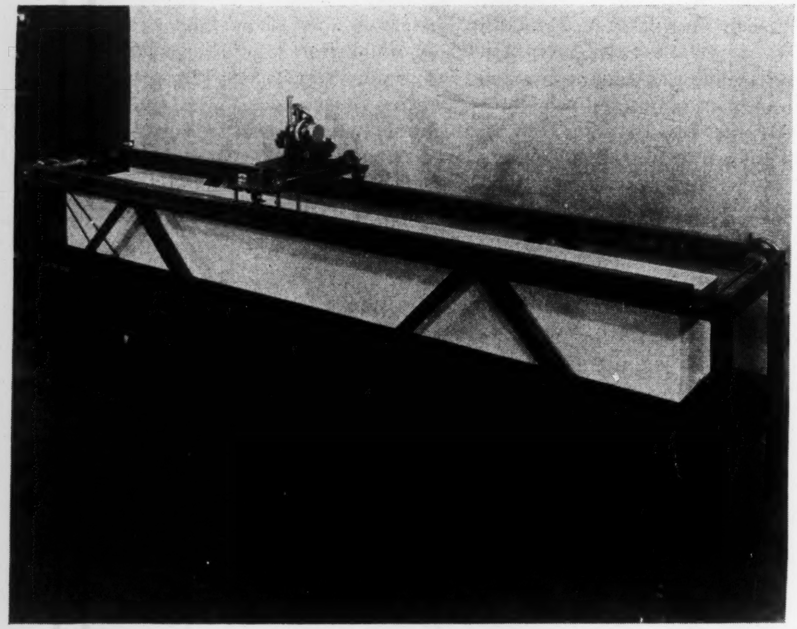

FIG. 1.—Scanner Assembly.

provided with two V-shaped wheels and one flat wheel. This insures that the transducers will move uniformly with respect to the specimen. The transducer carriage is moved along the rails by means of a chain drive, driven by a $\frac{1}{4}$-hp motor through a variable speed transmission. This motor is referred to as the translational drive motor. A clutch arrangement is provided to disengage the drive motor for manual scanning. The drive chain also engages a Selsyn master motor. Microswitches for stopping and reversing the drive motor at the end of a scan may be set to any desired position along the track.

The transducer assembly is a brass yoke designed to hold the two crystals in a rigid position with the faces of the transducers parallel, as shown in Fig. 2. Adjustments in the position of the crystals can be made by means of set screws.

To obtain the maximum sensitivity and resolution, the ultrasonic beam should have as small a cross-section as possible. This can be done in three ways —by using transducers having a small diameter, by masking, and by lenses. The latter two methods have been used in the work reported here.

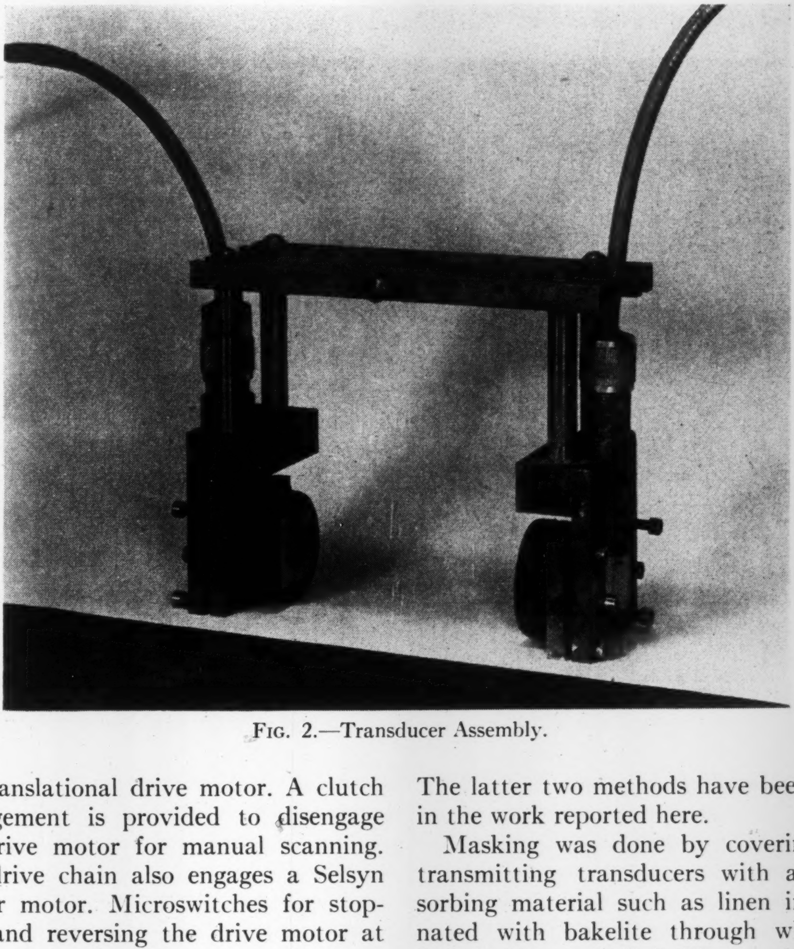

FIG. 2.—Transducer Assembly.

Masking was done by covering the transmitting transducers with an absorbing material such as linen impregnated with bakelite through which a hole had been drilled. The masks were made so that they could be slipped over the transducer and held firmly in place.

The contact lens technique was also used in this work, employing plano-spherical or plano-cylindrical lens, the plane side of the lens being fastened to the quartz transducer with wax. To minimize losses in transferring the ultrasonic energy from the transducer to the lens, the lens material should have an acoustical impedance as nearly equal to that of the transducer as possible. Since aluminum and quartz have nearly the same impedance, lenses made from aluminum were used. Aluminum also has a high acoustical refractive index when immersed in water (velocity of sound in aluminum divided by velocity of sound in water equals 3.5). The principles of physical optics carry over into the field of image formation with acoustic lenses. From the usual lens makers' formula, the radius of curvature can be calculated for a given focal length. A focal spot diameter of less than $\frac{1}{16}$ in. was observed at 5 Mc using a 1-in. square transmitting crystal. The lens on the receiving transducer was made spherically concave. For flat plate type specimens, masks have proven satisfactory. For cylindrical specimens, lenses have proven more satisfactory.

In addition to the transducers, a fixed-speed motor for moving the transducers up and down is mounted on the transducer carriage. The transducer assembly is connected to a sliding shaft which is in turn coupled to the elevating gear train. The vertical scanning rate can be changed by changing the elevating gear train. The amplitude of the vertical motion can also be changed by changing the gear train. A master Selsyn motor is also actuated by the vertical drive motor.

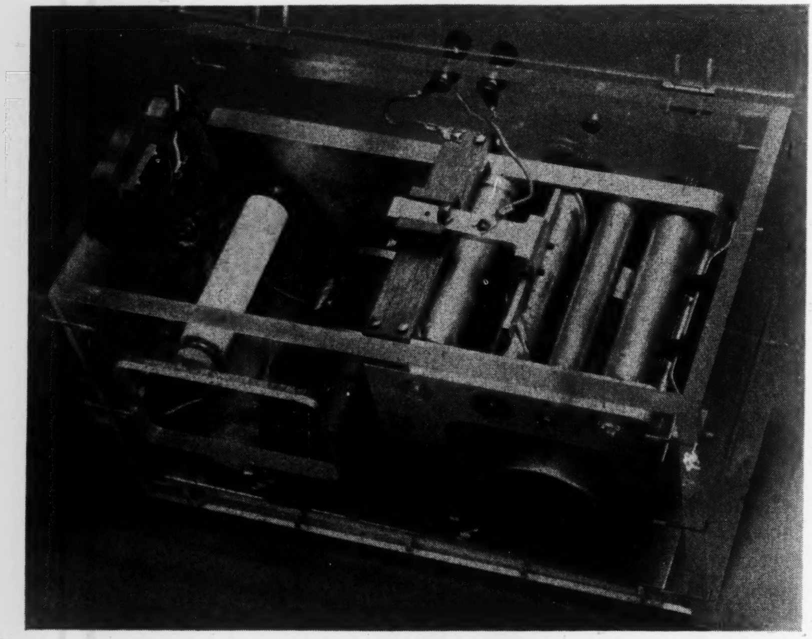

FIG. 3.—Electrosensitive Paper Recorder.

The scanner assembly can be used to test either flat or cylindrical specimens. When cylindrical specimens are being tested, the specimen is placed in a special rack in the tank and rotated in the couplant by means of a chain drive driven by a third motor through a variable speed transmission. In this application, the transducer carriage moves the transducer past the rotating specimen at a fixed elevation.

Recording System—The recording system consists of two parts: the electronic components and an electrosensitive paper recorder. The signal to be recorded is taken from the vertical amplifier of the Reflectoscope. The amplitude of this signal is a function of the ultrasonic transmission through the specimen—the better the transmission the greater the amplitude of this signal.

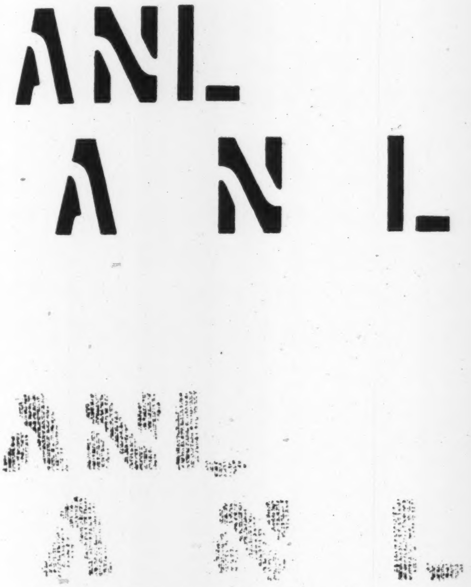

FIG. 4.—Electrosensitive Paper Record.

The electrosensitive paper record gives a permanent two-dimensional record of the ultrasonic transmission through the specimen being inspected. The recorder (Fig. 3) uses a moist electrosensitive paper which passes between two electrodes. The darkening of the paper is a function of the current flowing between the electrode and the writing speed. The greater the current flow the greater the darkening of the paper. The faster the writing speed the greater the current needed for satisfactory recording. Areas of good transmission appear as dark areas on the electrosensitive paper record, while areas giving poorer transmission appear as lighter areas. The electrosensitive paper must be moist for recording. The paper is sealed in plastic bags at the factory to retain moisture during storage. The paper is placed in a humidor in the recorder to prevent the paper from drying out while being used. Two kinds of electrosensitive papers have been employed, Alfax and Mufax.[2]

Two electrodes are used in the recorder: a fixed electrode which is the positive electrode and a movable electrode which is the negative electrode. The positive electrode is made from a strip of stainless steel $\frac{3}{64}$ in. wide and equal to the width (or circumference) of the specimen being tested. The negative electrode is a cylindrical drum on which is wound a single turn of nichrome wire to form a helix. As the moving electrode revolves, the point of intersection between the fixed electrode and the helix travels in a straight line always starting at the same edge of the paper and sweeping across it. By proper selection of the diameter of the recording helix, the width of the recording is equal to the width (or circumference) of the specimen.

A slave Selsyn motor actuated by the master Selsyn connected to the translational drive of the scanner is used to drive the paper feed in the recorder. The length of the recorder trace is equal to the length of the sample being scanned. The two Selsyn motors are connected in such a manner that, regardless of the direction of scanning, the recording paper is always moving in the same direction. As mentioned previously, a master Selsyn motor is connected to the two drive motors which are used either to move the transducers vertically or to rotate the sample in the tank. These master Selsyn motors are connected to the Selsyn motor which drives the recorder helix. A switch on the recorder permits the operator to select the proper master Selsyn. When scanning in the vertical direction, a cam, connected to the vertical drive motor actuates a microswitch arrangement so that the signal is applied to the recording helix only during the upward motion of the transducers. Figures 4 and 5 show typical recorder traces and the kind of sensitivity obtainable with this recorder using artificial defects. The upper half of Fig. 4 shows a stencil from which the letters ANL had been cut. The lower half of this same figure shows how the electrosensitive paper recorder reproduced the contour of the letters. The encircled area in Fig. 5 shows the record produced by an artificial defect $\frac{1}{8}$-in. in diameter. Experiments have shown that the electrosensitive paper record accurately indicates the shape, size, and location of actual nonbond or defective areas.

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[2] Alfax paper is manufactured by the Alfax Paper and Engineering Co., Westboro, Mass. The Mufax paper is manufactured by Muirhead and Co., Ltd., Beckenham-Kent, England.

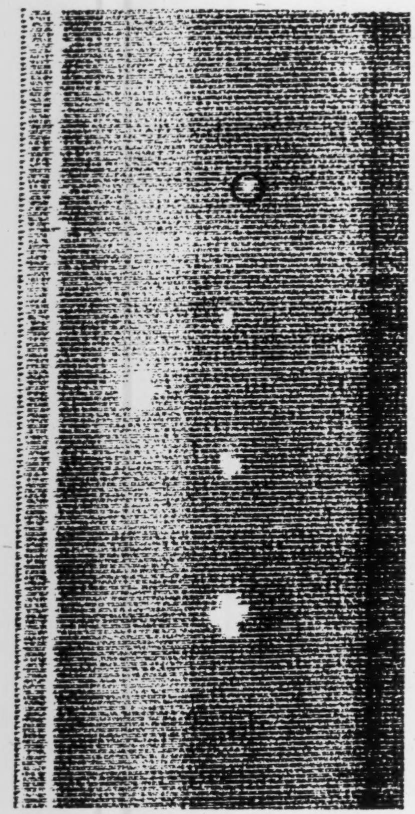

FIG. 5.—Electrosensitive Paper Record.

The electronic component of the recording system operates as follows: A negative keying pulse is taken from the synchronizer of the Reflectoscope. Because of the pulse delay in the Reflectoscope, the negative keying pulse precedes the pulse from the ultrasonic pulse generator. The keying pulse activates a gating circuit so that the initial pulse (which carries no intelligence concerning the test specimen) is gated out from the recorder. The ultrasonic pulse transmitted through the specimen opens the electronic gate, which allows the intelligence carried by the ultrasonic beam to be passed on to the recorder. It is necessary to rectify the signal before it is applied to the electrosensitive paper. A discriminator is provided so that noise and other extraneous signals may be rejected. A control is also provided which permits the operator to vary the contrast on the electrosensitive paper.

In order to assure that the defects were not present in the core material, the billets used in making the core were also tested for pipe, porosity, and blowholes. Figure 6 shows a typical electrosensitive paper record made of a large flat casting. This casting was so large that it was necessary to cut the casting into sections for testing. The composite record shown in Fig. 6 was made from the individual records. The light areas represent poor

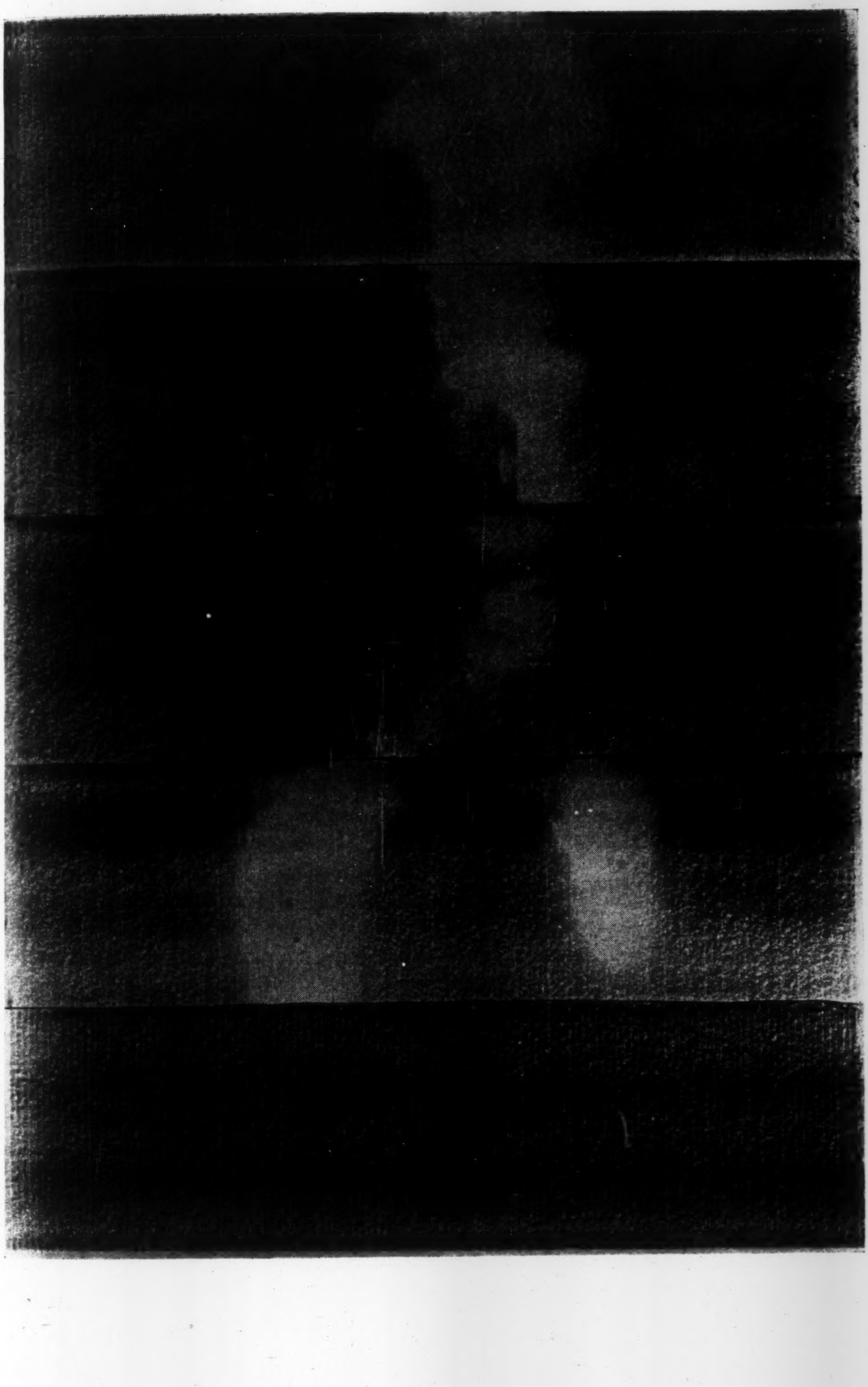

FIG. 6.—Electrosensitive Paper Record of Core Billets.

transmission, showing the presence of pipe, porosity, and blowholes, and the dark areas are areas of good transmission. In the testing a casting has been picked arbitrarily, after due destructive and nondestructive testing, as a standard casting, which is run periodically to check on the sensitivity and resolution of the equipment and to be sure that all the electronic components are working properly.

It is possible to use the transmission technique not only to determine lack of bond but also to determine the relative degree of bonding of zirconium-clad specimens. By proper selection of the amplification and recording level, the relative darkening of the paper gives a relative measure of the bond strength (Fig. 7).

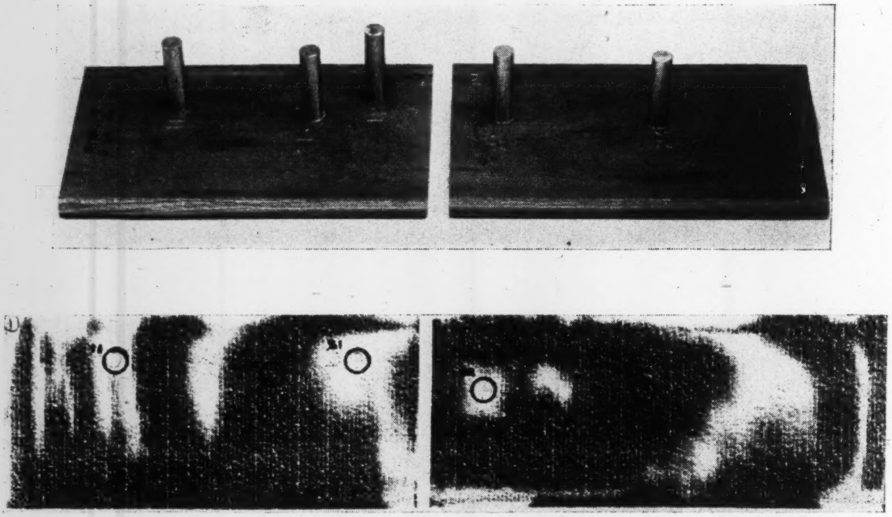

Fig. 7.—Zirconium-Clad Plate—Electrosensitive Paper Record and Percussion Welded Studs.

Bond strengths were determined also by tension testing, as follows: Zirconium studs $\frac{1}{4}$ in. in diameter and approximately 1 in. in length were percussion welded to the specimen, as shown in the upper half of Fig. 7. The area around each stud was machined through the cladding, leaving only the bonded area under the stud. The sample was then put in the tension testing machine and pulled. Comparison of the tension test with the relative darkening of the recorder trace gives a good correlation between bond strength and darkening of the paper.

Conclusion

The scanner and recorder described here have been successfully used for locating nonbonded areas in plate and cylindrical specimens. In a program for testing cylindrical type specimens, a translation speed of 8 in. per min and a rotational speed of 128 rpm was used. Unbonded areas of 0.03 sq in. or larger were found. In a program for plate type specimens, a linear scanning speed of 4 in. per min and vertical scanning speed of 480 in. per min was used. These scanning speeds give an overlapping of the vertical scans. At these scanning speeds, a pulse repetition rate of 120 per sec has proven satisfactory. A frequency of 5 Mc was used and the cross-section of the beam was $\frac{5}{64}$ in. Unbonded areas $\frac{1}{8}$ in. in diameter or larger can be found using this technique. At slower scanning speeds and smaller cross-section beams, defective areas less than $\frac{1}{8}$ in. in diameter can be found. However, in an actual testing program a practical compromise must be made between the time required for scanning a large number of specimens and sensitivity and resolution which can be achieved.

DISCUSSION

Mr. William R. Lorman.[1]—Have the authors considered that this apparatus with proper modifications may be of value in determining the internal condition of portland-cement mortars or concrete test prisms?

Mr. W. J. McGonnagle (*author*).—All our work has been with metals. In the Metallurgy Division at Argonne National Laboratory we do not have much contact with concrete. The Argonne National Laboratory is a reactor development center and the only use for concrete around the laboratory is for shielding purposes. However, I know that work has been done using ultrasonics to check the internal conditions of concrete.

Mr. Richard A. Proebstle.[2]—On the Zircalloy-II plate, can the width of the edge clad be detected? In other words with $\frac{1}{8}$-in. on the side of those Zircalloy-II plates would it be possible for the ultrasonic method to pick up the difference in density between the Zircalloy-II and the clad uranium?

Also, what were the two dark lines on each record that you had of the uranium casting? There were two dark lines on each record.

I would also like to know whether the entire subassembly can be tested.

Mr. McGonnagle.—It is possible to differentiate between the Zircalloy-II clad and the rest of the fuel element. We did not set the equipment up in this particular case for locating the Zircalloy on the side of the specimen but it can be done. You have at your control let us call it "contrast sensitivity." It is generally true in any nondestructive test that it is best suited to detect one particular thing under a certain set of conditions. In this particular case we were trying to detect lack of bonding and we set the equipment for maximum sensitivity for detecting lack of bond.

When you go from the specimen which in this case was a fuel element into water couplant the transmission through the water is very much higher than through the metal. The specimen is set so that it is above the bottom of the tank on a jig. The transducers actually go over the edges some little distance. The two white lines you see are due to the water.

The black lines are caused by a dull pen. The pen needs to be kept sharp. The paper, even though damp, will dull the pen. Sometimes the pens get a little flat spot on them and will cause a dark spot. The dark lines appear in every one of the figures and they occur at the same spot.

In answer to the third part of the question, from our experience I would say no. From a sonic point of view uranium is a good absorber of ultrasonic energy. We have used 5 Mc in testing. If you have thin specimens of uranium you can perhaps go up to frequencies of 10 to 12 Mc. When testing a subassembly you get the loses at all the interfaces as well as the absorption in the material itself. You lose energy at every interface, so after several interfaces you have lost much of your energy and intelligence. If you found a defect in a subassembly using low frequency ultrasonics, there would be the problem of knowing in which component of the subassembly the defect occurred. One of the disadvantages of using the transmission technique is that one cannot locate exactly in depth where the flaw is. As far as we were concerned we were looking for lack of bonding and any specimen which had a lack of bonding was rejected.

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[1] Materials Division, U. S. Naval Civil Engineering, Research and Evaluation Laboratory, Construction Battalion Center, Port Hueneme, Calif.

[2] Aberdeen Proving Grounds, Aberdeen, Md.

MR. LEO SOFFA.[3]—What does this do that other ultrasonic equipment that is on the market does not do?

MR. MCGONNAGLE.—Most ultrasonic equipment is set up for reflection type of work, but in thin samples you cannot use the reflection type of technique. You have to use a transmission type of technique.

However as a complete system it has some unique features. The attribute that is most outstanding is that we are producing a permanent two-dimensional picture right in front of your eyes, so to speak, without further developing or other processing. That is probably the outstanding virtue of this scanning and recording system.

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[3] Northrop Aircraft Inc., Hawthorne, Calif.

INELASTIC DEFORMATION OF NYLON UNDER COMPRESSION

By C. C. Hsiao[1]

Synopsis

The effect of rate of straining on compressive stress-strain properties of nylon at room temperature has been studied. An inexpensive apparatus with a simple method for testing at moderate rates of straining has been used, and many interesting properties such as the transitional stress-strain behavior from elastic to inelastic, the variation of volume and the inelastic deformation as well as the recoverable ability of the material have been observed and reported.

At constant strains, the effect of log of strain rate on loading is found to be linearly related within the range of rate of straining investigated. If this linear relationship is extended, stress-strain properties at fairly low rates of straining can be extrapolated and long-time creep or relaxation characteristics of the material at high stress levels and large deformations may be derived. The possibility of describing the flow properties of the material by a family of equations is discussed. It seems that the mechanism of the flow behavior of nylon under compression may be interpreted by the Eyring molecular reaction rate theory.

In recent years the simultaneous rapid growth of the plastics industry and the intensive applications of its various products has led to the importance of both theoretical and experimental investigations on the mechanical properties of polymeric materials. Most of these materials are sensitive to temperature and time and this is particularly true of linear high polymers where the changes due to different rates of straining even at room temperature conditions are sufficiently significant to warrant careful investigation, (**1**, **2**).[2]

One of a number of well-known linear polymers widely used in various applications is nylon. Of this material, the temperature stability, high strength, and low coefficient of friction as well as the remarkable ability of recovery contribute to its service application. When used under high compressive loading, one of its useful applications, for instance, is the elastic stop nut in which the nylon collar prevents the nut from loosening.

In order to explore the effect of variable rates of straining upon the compressive stress-strain properties, a commercial grade of nylon, type FM-10001, was studied. A series of simple compression tests was carried out to large deformations at a constant room temperature.

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[1] Associate Professor, University of Minnesota, Minneapolis, Minn.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, p. 1436.

A number of interesting results were observed and are reported in this paper. Along with this investigation, an attempt was also made to predict the long-time creep and relaxation behavior of the material from the test results.

EXPERIMENTAL

Apparatus, Specimen, and Test Procedure:

All the tests were carried out with the use of a small Dillon tester in a constant temperature and humidity room. The chief components of the control system for maintaining a constant strain rate throughout a test were a π gage, a rotating disk with mounted resistance wire, and a strain indicator as described in a previous paper (2). Figure 1 illustrates schematically the general experimental setup.

As a result of several preliminary tests, a cylindrical specimen of $\frac{3}{4}$ in. by $\frac{1}{2}$ in. diameter was selected. Specimens were machined from a commercial grade of molded nylon rod, type FM-10001, of $\frac{5}{8}$ in. diameter. All specimens were conditioned in a constant-temperature (77 ± 2 F) and constant-humidity (50 ± 2 per cent RH) room for many days prior to testing.

In carrying out each test, the two knife edges of the π gage were secured to the specimen separately by elastic threads, at a desired gage length of about $\frac{1}{4}$ in. A thin layer of petroleum jelly was applied to the two end surfaces of the cylindrical specimen before the compressive loading was applied. The two-wire resistance gages cemented on the π gage and the resistance wire on the rotating disk were so connected to the strain indicator that the total resistance in the circuit was always kept constant for the duration of the test. When the resistance wire on the rotating disk was turned by a timing motor at a predetermined rate, the corresponding rate of strain change of the specimen was followed by manually applying sufficient compressive loading. Fairly accurate data such as load, time, and diameter of the specimen were simultaneously

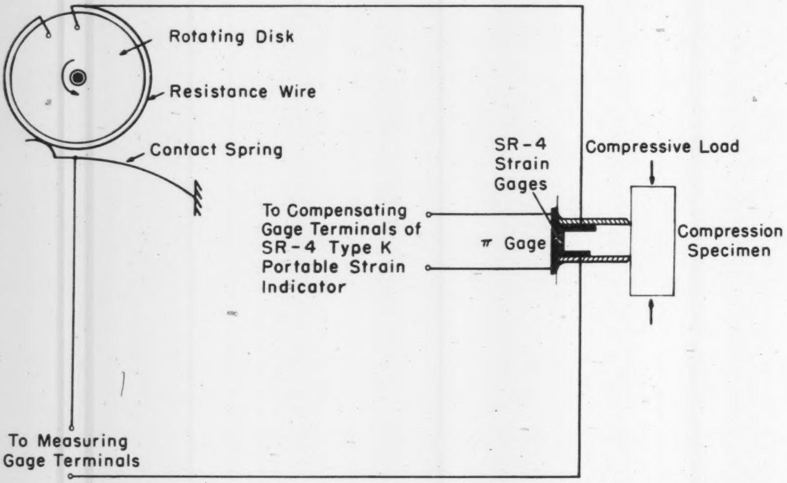

FIG. 1.—Schematic Diagram of the Experimental Setup for Constant Strain Rate in Compression Test.

recorded for the range of strain rate investigated.

Test Results and Correlation:

Simple compression tests of nylon were run on a number of specimens at a range of strain rate from 12 × 10^{-6} per sec to approximately 712 × 10^{-6} per sec. A detailed plot of a typical compressive stress-strain curve for a strain rate of about 12 × 10^{-6} per sec is shown in Fig. 2. There are three stress-strain curves plotted for this single test. The nominal stress-strain curve is represented by the solid line drawn through the actual data of open circles. The crossed points are those experimental true stress points calculated at various strains, and the plot of this true stress-strain curve thus obtained is shown by the broken line. It will be noted that there is little difference between the nominal and the true stress until shortly before the 3 per cent first transitional strain change is reached, then the true stress falls increasingly below the nominal stress due to continued increase in cross-sectional area. However, there was no bulging observed within the gage length used until the specimen is strained to a value

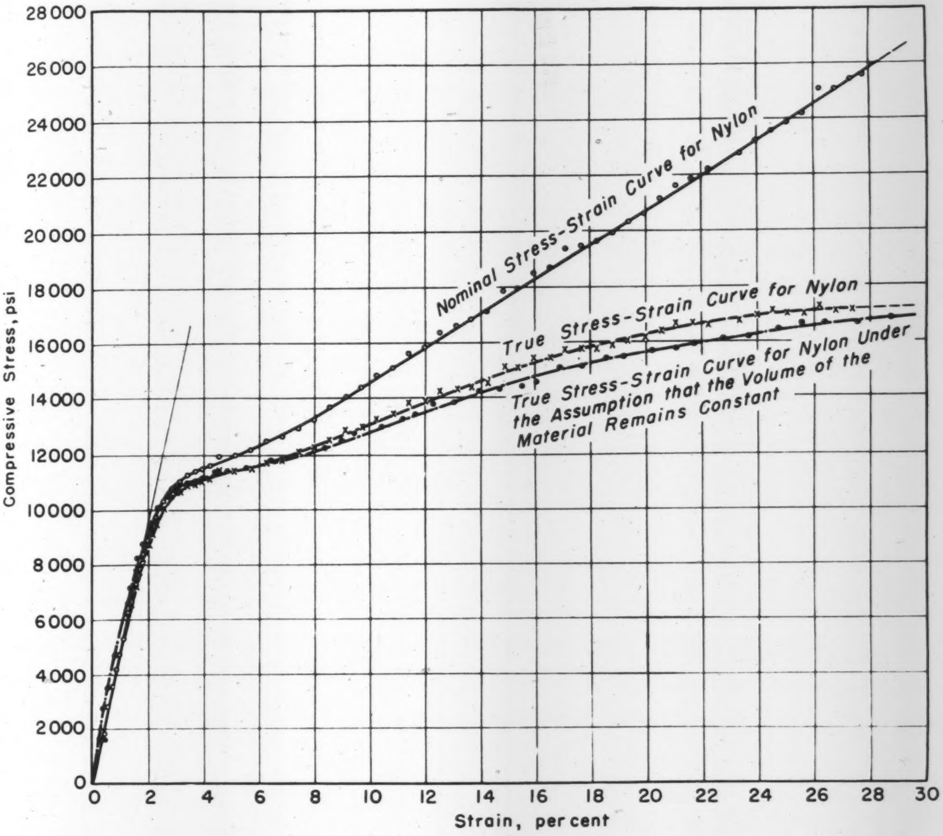

FIG. 2.—Compressive Stress-Strain Curves of Nylon at 11.9 × 10^{-6} per sec Rate of Straining.

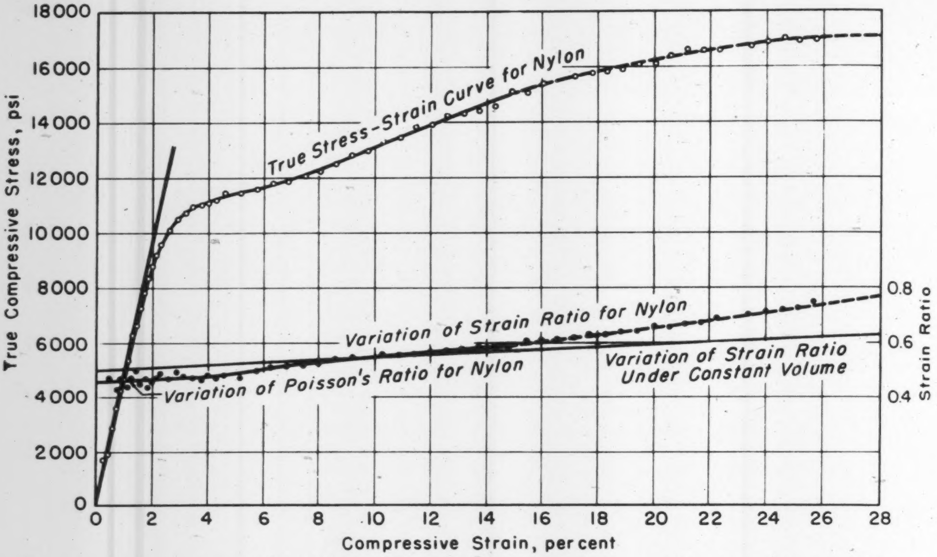

FIG. 3.—Compressive True Stress-Strain Curves and Variations of Strain Ratio for Nylon at 11.9 × 10⁻⁶ per sec Rate of Straining. Size of Specimen $\frac{3}{4}$ by $\frac{1}{2}$ in. diam. Gage Length of Specimen $\frac{1}{4}$ in.

of about 15 per cent. This effect of bulging was observed by comparing the gage section of the specimen and a highly polished flat surface. The change in its diameter was also measured from time to time. The accuracy of the measurement was within 0.001 in.

If these instantaneous true stresses are kept and their corresponding longitudinal strain values are calculated from the measured transverse strain data with the assumption that the volume of the material remains constant throughout the duration of test, then the true stress-strain curve following the solid circles can be obtained. This curve is not very much different from the true stress-strain curve just described. But the interesting information here lies in the fact that the initial portion of this stress-strain curve does not coincide with that of the other two for which the strain quantities are actually measured. This deviation may be attributed to the volume change of the material in its initial elastic region.

The inclined thin line extending out from the initial portion of the true stress-strain curve gives the constant ratio of

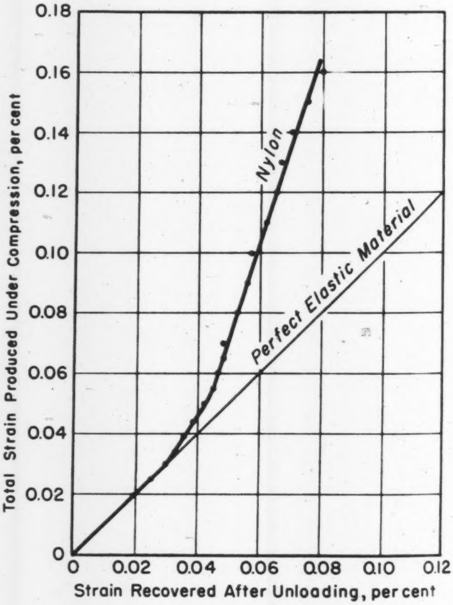

FIG. 4.—Strain Recovery of Nylon.

stress to strain which is ordinarily termed modulus of elasticity. The value of this modulus of elasticity of the material tested at 11.9 × 10^{-6} per sec rate of straining is 4.8 × 10^5 psi. This value remains the same until about 6000 psi of stress is reached. Above this point, the tangent modulus gradually decreases from the value of elastic modulus. Finally, beyond about 6 per cent strain change, the transitional behavior seems over, the tangent modulus becomes constant again. For the nominal stress-strain curve the value is 0.38 × 10^5 psi within the strain range from 6 per cent to 15 per cent. Beyond this upper limit of 15 per cent strain change, the specimen bulges, which makes significant interpretation difficult.

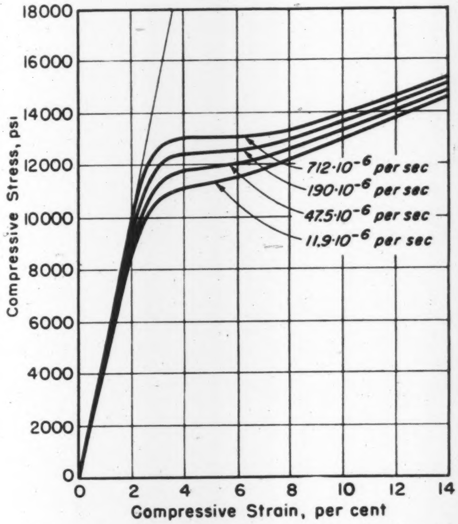

FIG. 5.—Compressive True Stress-Strain Curves of Nylon at Various Constant Rates of Straining.

In order to show the variation of the lateral and longitudinal strain ratio of the material under compressive loading, the true stress-strain curve was replotted in Fig. 3. Corresponding to almost all the experimental points, values of the strain ratio are determined from direct measurements and are shown as solid dots. Within the elastic range of the material under a simple state of stressing, this linear lateral and longitudinal strain ratio is essentially a constant and is generally termed Poisson's ratio. Beyond the elastic range, this strain ratio varies gradually according to not only the volume change but also the physical dimensional change of the material. In order to determine whether the variation of the strain ratio is associated with the volume change of the material, a reference curve is given of the variation of the strain ratio for a constant volume. By considering a unit volume under compression, if ϵ is the longitudinal strain change and ν the ratio of the linear lateral strain and the linear longitudinal strain, then

$$(1 - \epsilon) \cdot (1 + \nu\epsilon)^2 = 1$$

Thus the variation of the strain ratio, ν, may be expressed in terms of the longitudinal strain change in the following manner:

$$\nu = \frac{1}{\epsilon}\left(\frac{1}{\sqrt{1 - \epsilon}} - 1\right) \qquad (0 < \epsilon < 1)$$

Using the same scale as shown at the right-hand side of Fig. 3, the variation of the strain ratio as a function of longitudinal strain change is drawn as a thin line. Comparing with the measured strain ratio, it seems that a change in volume of the material occurred during testing. Since no bulging of the specimen was observed below 15 per cent strain change, it appears that the volume of the material first decreases as the stress increases and then increases as the stress is well over 12,000 psi. While there is little doubt that this interesting phenomenon is associated with the variation of the molecular structure of the material, there is no satisfactory explanation to account for the change.

Also shown in Fig. 3 are two thin inclined lines. One of the two lines indicates that the modulus of elasticity of the material tested at a constant strain rate of 11.9 × 10^{-6} per sec is about 4.8 × 10^5 psi. The other inclined thin line having a slope of about 5.0 × 10^5 psi indicates that the modulus of elasticity increases slightly when the material is tested at a higher strain rate of 712 × 10^{-6} per sec. This effect will be described in greater detail later.

In order to determine the ability of the material to recover, a fairly rapid cyclic loading and unloading test for a similar compression specimen was conducted (Fig. 4). A strain change was first produced under compressive loading in a few seconds and was then recovered either entirely or in part after rapid unloading until no appreciable amount of strain change was observed. This procedure was repeated until the transitional behavior was believed to be over. It is interesting to note that below 3 per cent strain change, the recovery is 100 per cent. Above this point a permanent set of about 38 per cent of any additional strain change was recorded which means that the total recoverable strain change is 100 per cent × 3 per cent + 62 per cent of any additional strain change produced. This is true until the material reaches another transitional strain value of about 5½ per cent. As they have occurred in the simple compression test, again, there are two transitional strains observed in this test. Are these transitions due to molecular flow or to structural changes of the material? Perhaps the study of this transitional behavior by means of proper physical measures will be of great value to the understanding of this interesting mechanism.

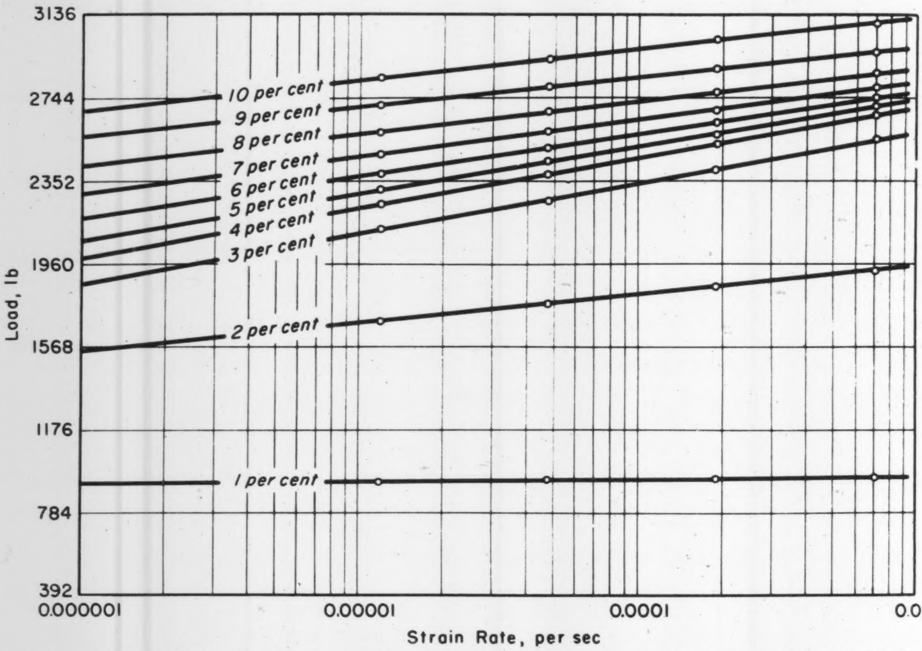

FIG. 6.—Effect of Rate of Straining on Compressive Load for Nylon at Various Constant Strains.

The effect of rate of straining upon

the compressive true stress - strain properties of this material can well be illustrated by Fig. 5. In this figure a set of curves is plotted at four different values of strain rate ranging from 11.9 × 10^{-6} per sec to 712 × 10^{-6} per sec as indicated. The modulus of elasticity is seen to increase slightly with increase of strain rate.

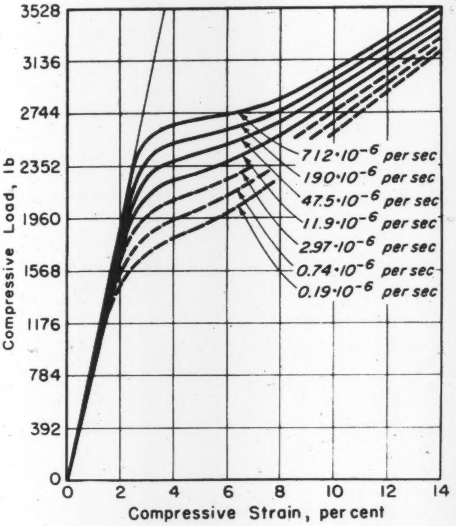

FIG. 7.—Compressive Load-Strain Curves of Nylon at Various Constant Rates of Straining.

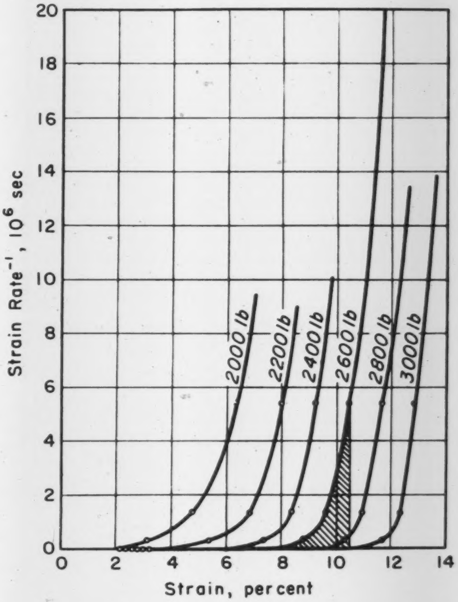

FIG. 8.—Compressive Strain *versus* Reciprocal of Compressive Strain Rate.

Under compressive loading, both the nominal and true stresses at any particular strain value increase with increase of strain rate. Data show that the increase of load is a linear one when plotted against log of strain rate as shown in Fig. 6. By extrapolation for a number of different strain quantities, load-strain curves at fairly low rates of straining can be constructed. These are shown in Fig. 7 in broken lines together with the four original tests run at moderate strain rates. From these curves, a replot of the reciprocal of strain rate against strain for any constant load values is possible. This is shown in Fig. 8. Then the shaded area, for example, in this figure represents the time required for the material to reach a 10.45 per cent strain change under a constant load of 2600 lb, provided that similar size of specimen is used. Thus for any given values of strain, the area under these curves will then give corresponding values of time so that a derived creep curve for any constant load may be plotted. Figure 9 shows several creep data thus derived for a load of 2600 lb which are in fairly good agreement with the experimental curve.

Similarly, in a slightly different manner, relaxation curves such as that in Fig. 10 can also be derived. However, no experimental verification was attempted.

It should also be pointed out that this procedure did not seem to apply well if the strain change was small. But for large deformations, where the configurational elasticity may be insignifi-

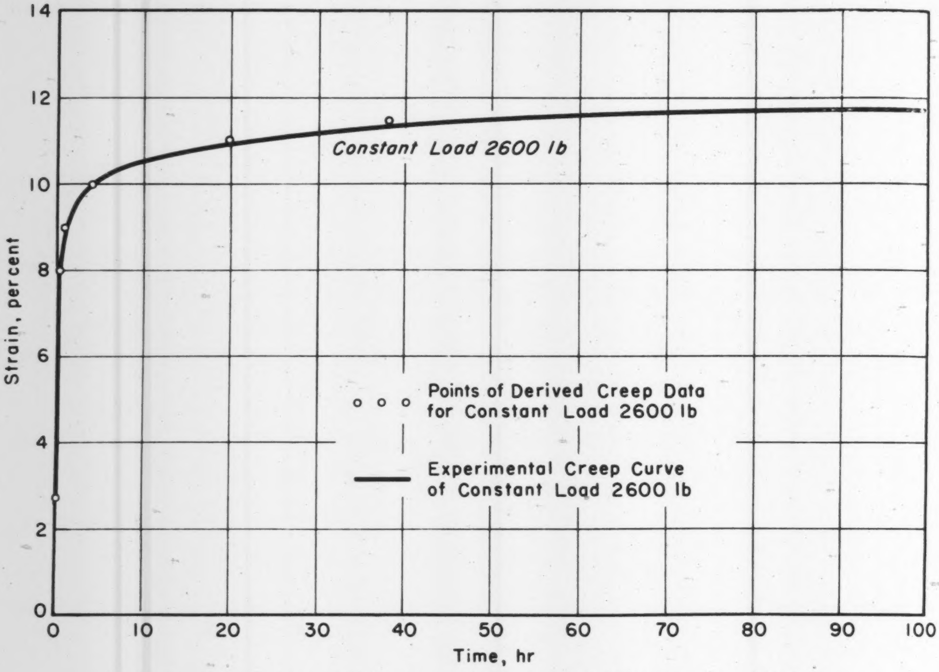

FIG. 9.—Comparison of Derived and Experimental Creep Curves of Nylon.

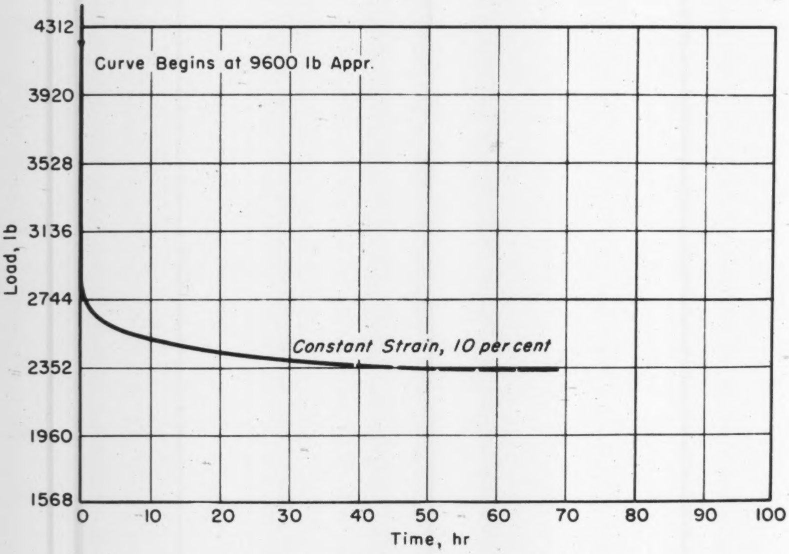

FIG. 10.—Derived Relaxation Curve for Nylon.

cant, an approximate correlation as demonstrated before is possible.

DISCUSSION

The concept of the existence of a mechanical equation of state for solids has been studied for quite some time. It has been proposed (**3**, **4**, **5**, **6**), under certain conditions and approximations, that stress, σ, for plastic flow be considered a function only of the instantaneous values of plastic strain, ϵ, strain rate, $\dot{\epsilon}$, and temperature, T: $\sigma = f(\epsilon, \dot{\epsilon}, T)$. One of the assumptions is that prior strain history has no effect on the subsequent mechanical behavior of the solid. Another assumption is that time is not explicitly involved as a separate variable. If these conditions are met, then tensile or compressive creep and relaxation properties of the solid can be derived from simple tension or compression behavior under various constant rates of straining. The required procedure is simply to use the stress-strain data at varying strain rates to plot graphs of the reciprocal of strain rate *versus* the strain. For given values of strain, the area under these curves will then give values of time so that a derived creep strain-time curve can then be plotted. Similarly, a derived relaxation curve can also be obtained for any constant values of strain change.

In order to see whether some such relation as the above would be a good approximation for plastic materials and whether the concept could be employed to predict creep and relaxation behaviors, an attempt was made to compare the derived compressive creep curve with experimental data. It seems that a fair agreement is obtained at high stress levels and large deformations. Unfortunately, sufficient experimental data are not available for a more conclusive comparison.

In general, it is difficult to prove the validity of the mechanical equation of state for solids, as structurally they are more or less discontinuous media. The variation in the structure of a material under any loading condition would tend to invalidate any attempted correlation. For the present case, however, the simple correlation seems good. It may be that for large deformations under compressive state of loading, nylon behaves more closely to that of an ideal plastic material. Perhaps, an even better correlation may be obtained if the effects of structural orientation (**7**, **8**, **9**) as well as the factor of rate of stressing of the material can be taken into consideration.

One important aspect of this consideration is that stress is an active quantity while strain and strain rate are passive. As a result of stressing, strain, ϵ, rate of straining, $\dot{\epsilon}$, and perhaps acceleration of straining, $\ddot{\epsilon}$, will occur and will govern both the flow properties and the structural variations of the material. This is true for a material or a system of molecules subject to a constant stress, σ. For many practical cases, usually the rate of stressing, $\dot{\sigma}$, and perhaps a varying rate of stressing, $\ddot{\sigma}$, are involved. Under these conditions, it would seem necessary to take at least the stress rate, $\dot{\sigma}$, into consideration when a material is subject to stress.

Despite the extremely complicated flow phenomena of solid high polymers under stress, some systematic analysis of the flow properties for nylon can be made empirically. Referring to the compressive stress-strain curves in Fig. 4, if log of strain rate is plotted against the compressive stress for constant values of the compressive strain as shown in Fig. 11, a linear relationship between the stress, σ, and log of strain rate, $\dot{\epsilon}$, is realized. If again, we assume that this linear relationship can be extended indefinitely, we may represent this family

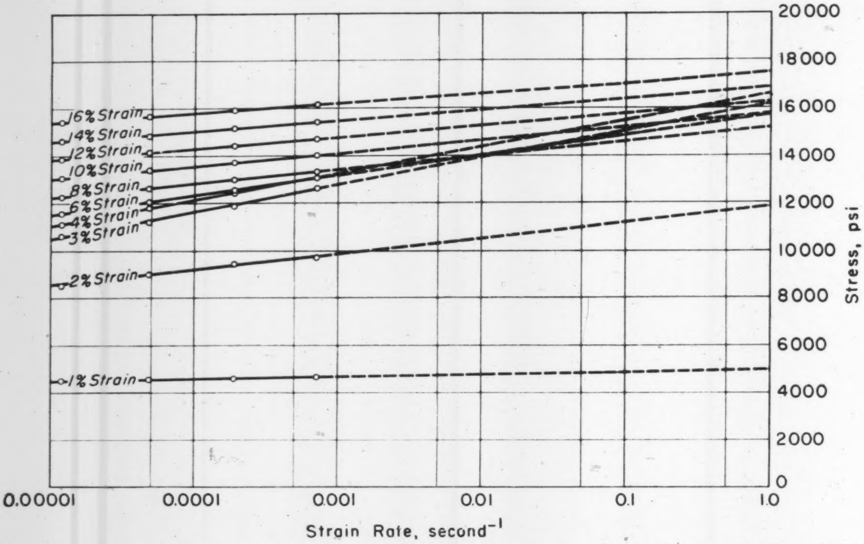

FIG. 11.—Effect of Rate of Straining on Compressive Stress for Nylon at Various Constant Strains.

of straight lines in the following manner:

$$\sigma = b_i + m_i \log \dot{\epsilon}$$

where b_i and m_i are constants depending upon the values of strain, ϵ, and temperature, T. This seems to have a proper physical significance if a molecular theory of flow is considered.

According to the Eyring molecular reaction rate theory (**10**) the viscous flow process is essentially the consequence of the movement of a molecule or a segment of a molecule from one equilibrium position to another across a potential energy barrier. If E is the value of the potential energy to be overcome, the frequency with which such steps occur under the influence of the thermal agitation is proportional to $e^{-E/RT}$. When a stress, σ, is applied to the system, owing to the modification of the energy barrier, the net rate of flow, $\dot{\epsilon}$, becomes:

$$\dot{\epsilon} = 2ke^{-E/RT} \sinh \frac{c\sigma}{RT}$$

where k and c are constants associated with Planck's constant, molar volume, Avogadro's number and molecular distances. For solid polymers, relatively large forces may be applied and $c\sigma$ may be very much greater than RT. Then the above flow equation may be reduced to:

$$\dot{\epsilon} = ke^{-E/RT}e^{\frac{c\sigma}{RT}}$$

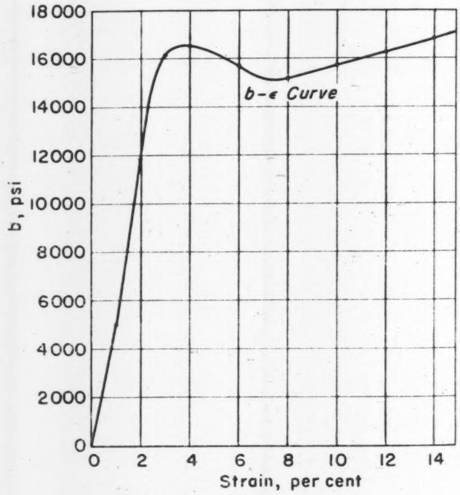

FIG. 12.—b-ϵ Curve.

or

$$\sigma = \frac{E - RT \ln k}{c} + \frac{RT}{c} \ln \dot{\epsilon}$$

The remarkable resemblance between this theoretical equation and each of the family of experimental curves

$$\sigma = b_i(\epsilon, T) + m_i(\epsilon, T) \log \dot{\epsilon}$$

is clearly shown. The theory is derived according to the molecular movement of a medium at one structural state under stress. The experimental curves indicate that perhaps there exists a continuous variation of the structure undergoing, however, essentially the same mechanism of molecular movement. Aside from the material constants, this flow mechanism is also dependent upon temperature and strain change.

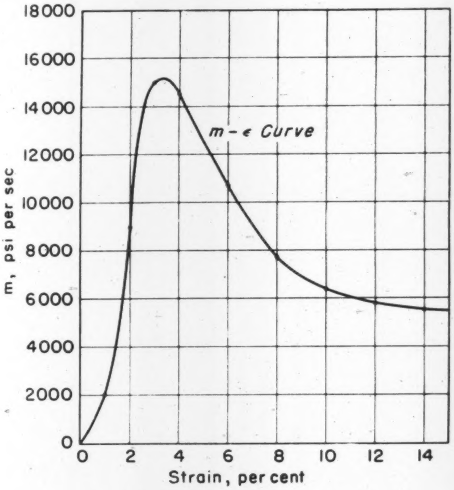

FIG. 13.—m-ϵ Curve.

For the purpose of obtaining a general idea as to the relationship between b and ϵ as well as m and ϵ for nylon, two individual curves are constructed from Fig. 11. The b-ϵ curve is determined by plotting the strain values against their corresponding intersections with the vertical line $\log \dot{\epsilon} = 0$. This curve is shown in Fig. 12. The m-ϵ curve is constructed by plotting against values of strain, ϵ, the slope of each corresponding line. Figure 13 shows the general relationship between m and ϵ.

It might be of interest to point out that the b-ϵ curve becomes an inclined straight line after the transitional behavior is over and the m-ϵ curve tends eventually to level off. In other words, when the material is at its steady state of flow, b and ϵ are linearly related at room temperature and m tends to be independent of ϵ. Under these conditions, if the value of strain rate is not zero, then the family of equations may be reduced to the following simple form:

$$\sigma = \alpha + \beta\epsilon + \gamma \log \dot{\epsilon}$$

where α, β and γ are constants.

CONCLUSION

From the experimental results on the inelastic deformation of nylon by compression at constant temperature, a family of empirical stress-strain relationships are obtained to describe the mechanical behavior of the material. The fact that each of the family of flow equations has essentially the same form as that given by the molecular reaction rate theory is quite significant. It appears that, as a result of stressing, a continuous variation of the molecular structure of the material exists. However, beyond the transitional region, a steady state of flow may be present at high stress levels. An extension of this study within the general framework may reveal further valuable information regarding the viscoelastic behavior of polymeric solids. A natural outgrowth of the work would be in the investigation of the stress-strain laws at varying strain rates and temperatures as well as, perhaps, varying stress rates for polymeric materials. Of course, a better understanding would result if the structural information of the material is simultaneously obtained with the stress-strain data.

Acknowledgment:

The author deeply appreciates the advice and counsel given by J. A. Sauer of the Pennsylvania State University.

It is also a pleasure to express thanks to B. J. Lazan and W. M. Warner of the Department of Mechanics and Materials of the University of Minnesota for their helpful comments.

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A METHOD FOR MEASURING SURFACE FLAMMABILITY OF MATERIALS USING A RADIANT ENERGY SOURCE*[1]

By A. F. Robertson,[2] D. Gross,[2] and J. Loftus[2]

Synopsis

There has been a need for a relatively simple yet sufficiently severe and reproducible method for measuring the surface flame spread properties of materials. The present paper describes a test method which has been developed to meet this need. The method employs a radiant heat source consisting of a 12 by 18-in. panel in front of which a 6 by 18-in. specimen of the material being tested is mounted. The specimen orientation is such that ignition is forced near the top and the flame front progresses downward. The rate of progress of the flame front together with a factor involving the rate of heat generation by the material under test are combined by means of an empirical relationship to provide a flame spread index for the material. It is demonstrated that the test method is relatively insensitive to changes in ambient variables and modifications in mechanical adjustment. The classification of materials with respect to flammability follows an order which appears to be consistent with information currently available on their behavior during fires.

The rate with which an accidental fire will develop (in the absence of fire detection and fighting facilities) into a full fledged fire involving a whole building depends primarily upon three major factors: (*a*) the flame spread properties of the interior finish, (*b*) the extent of compartmentation and degree of ventilation, (*c*) the fuel contributed by and the manner of distribution of the building contents. It is only the first two of these over which the builder and approving agency have some control. In spite of a number of full-scale burn-out tests (**1, 2, 3**)[3] the complexity of the problem has prevented more than a rather crude appreciation of the relative importance of the factors governing growth and spread of fires and the most practical methods of their control. Because of this, rather arbitrary methods have been adopted for evaluating the flame spread properties of materials and for specifying the degree of compartmentation desirable. The present discussion will be confined to methods of measuring the flame spread properties of building materials.

Test methods for evaluating this property have been developed and studied both at the National Bureau of Standards and at other laboratories for many years. The Cocoanut Grove fire in 1942, however, was largely responsible for the impetus in adoption by Underwriters' Laboratories, of the tunnel test,[4] as the standard fire hazard test method used by their laboratory (4). The method developed appears to classify many materials in an order which empirical experience has shown to be desirable. The cost of the test equipment, together with the size of the test specimens, however, have delayed if not prevented its use by private manufacturers, testing, and government laboratories. Numerous test methods have been developed in which smaller test specimens are used. Examples of these include the flame resistance test method described in Federal Specification SS-A-118b,[5] the spread-of-flame test method of British Standard BS 476,[6] and the inclined panel test described in Federal Specification TTP-26a.[7]

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] A brief announcement of the development of this method has been reported in *Technical News Bulletin*, Nat. Bureau Standards, Oct., 1955.

[2] Fire Protection Section, Nat. Bureau Standards, Washington, D. C.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, p. 1453.

Federal Specification SS-A-118b was developed for evaluating certain combustible properties of acoustical tile but has frequently been proposed and used in the formulation of building codes. When used for this purpose it is a very severe test and classifies materials in only a qualitative fashion. The British test method was developed during the war as a result of their first-hand experience with fire. The method is deficient, however, in that it provides too mild a test as indicated by recent studies made at the Fire Research Station (5). The inclined panel test, intended for evaluation of paint finishes, also utilizes a comparatively mild fire exposure of the specimen, and consequently it has not generally been considered suitable for measuring the flame spread properties of materials.

Because of these difficulties it appeared desirable to attempt development of a new flame spread test method which would involve use of only modest size specimens, be relatively compact and inexpensive to reproduce and use, and last but not least, would measure the flame spread behavior of materials in a manner consistent with their observed performance during fires. This paper presents a description of the test method developed together with test data on representative building materials. Particular effort has been made to provide sufficient equipment design and calibration data to permit others to reproduce the test facility.

PRIMARY CONSIDERATIONS

During the flaming combustion of solids, the combustible gases which sustain the flaming are released from the solid as a result of heat received by the solid itself. This heat may be supplied by some external source, but in most cases where flaming occurs, it is largely supported by radiation from the flames and convection from the hot gases. The rate with which a flame can spread along the surface of a material with combustible content is thus largely determined by the rate with which its surface may be heated. The thermal properties and dimensions of the specimen as well as the size and character of the flames produced are therefore of considerable importance in determination of its flame spread behavior.

The wavering or turbulent characteristics of flames from a burning solid obscure the progress of volatilization of gases from the solid and consequently the spread of flame on its surface. In the past, it has therefore been considered desirable to use test specimens having at least one dimension large compared both to the length of the flames and to the fluctuation in flame length produced during burning. If, however, a controlled external source rather than flame radiation is used to supply heat to the nonflaming portion of the specimen, more accurate observations of the progress of the flame front are possible when using much smaller sizes of test specimens. Under these conditions, the rate of spread of this flame front does not include the heating effect that the flaming portion would normally contribute to the unignited portion. It is desirable, therefore, to devise some method for measurement of the heating power of the flames and for its incorporation with the rate of spread of flame measurement.

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[4] This test has also been incorporated as ASTM Tentative Method of Fire Hazard Classification of Building Materials (E 84 – 50 T), 1955 Book of ASTM Standards, Part 4, p. 1204.

[5] Federal Specification, Acoustical Units; Prefabricated, SS-A-118b, Aug., 1954, U. S. Government Printing Office.

[6] "Fire Tests on Building Materials and Structures," British Standard, No. BS 476, London 1953.

[7] Federal Specification, Paint Interior White and Tints, Fire Retardant, TT-P-26a, 13 Jan., 1954, U. S. Government Printing Office.

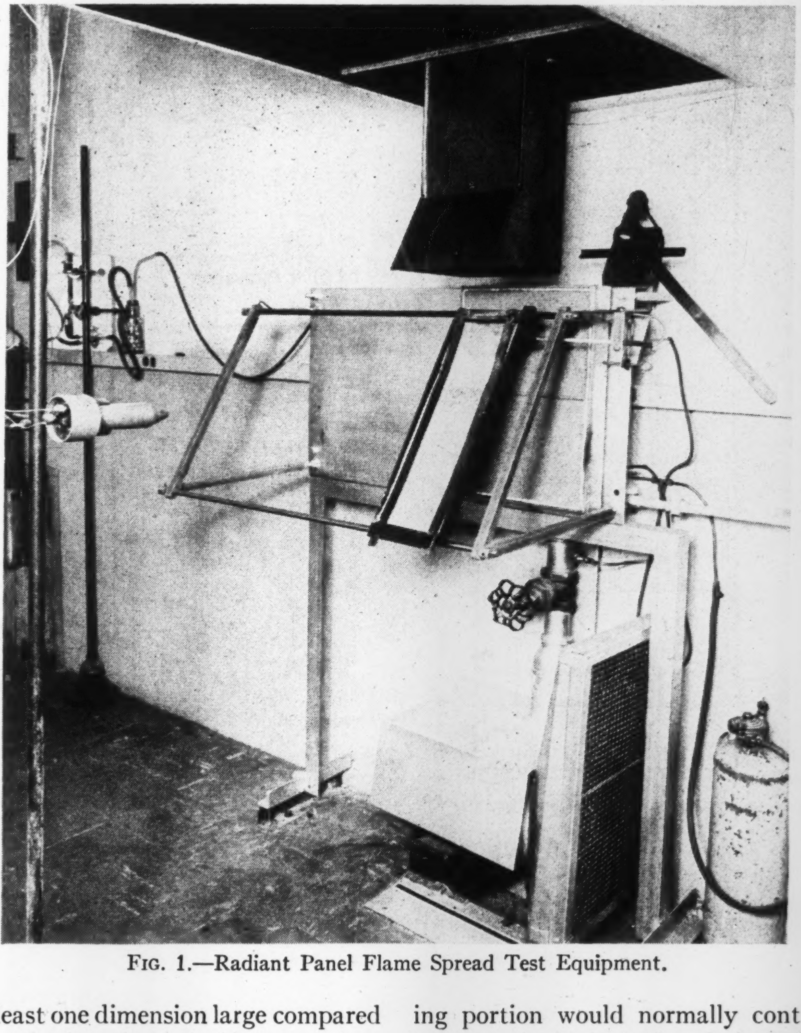

FIG. 1.—Radiant Panel Flame Spread Test Equipment.

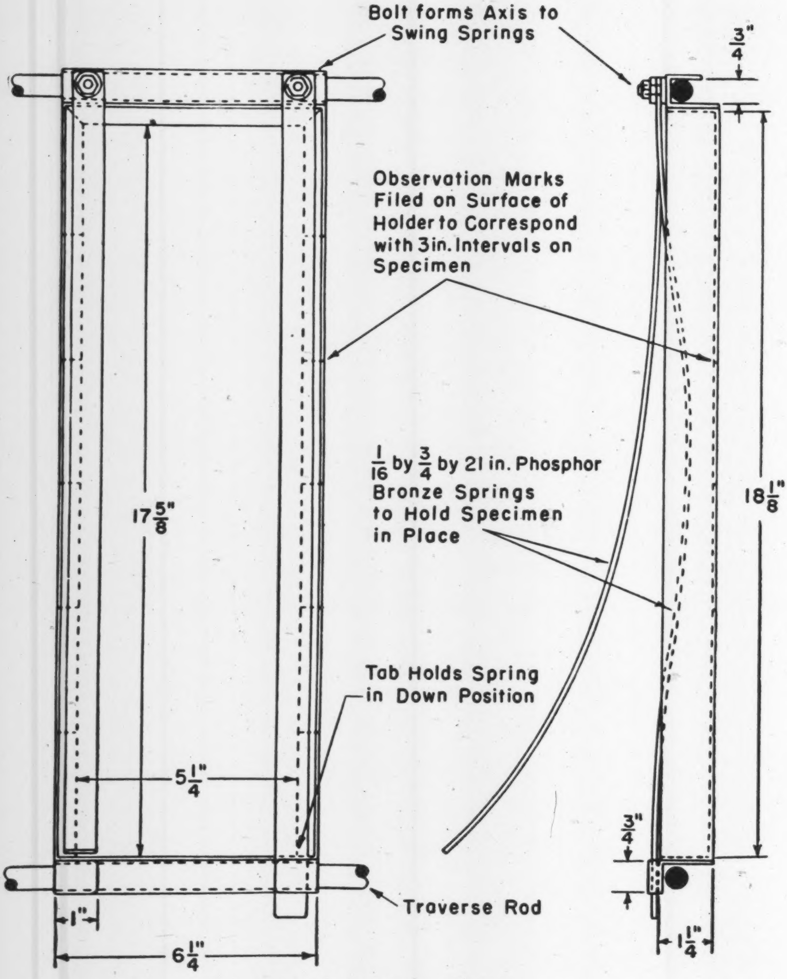
Bolt forms Axis to Swing Springs
3/4"
Observation Marks Filed on Surface of Holder to Correspond with 3in. Intervals on Specimen
1/16 by 3/4 by 21 in. Phosphor Bronze Springs to Hold Specimen in Place
17 5/8"
18 1/8"
Tab Holds Spring in Down Position
5 1/4"
3/4"
Traverse Rod
1"
6 1/4"
1 1/4"

FIG. 2.—Specimen Holder.

The apparatus to be described takes into consideration the factors mentioned above. The specimen is oriented in front of a radiant heat source in such a manner that ignition takes place at the top of the specimen and the flame front travels downward. Flames rise away from the unignited portions of the specimen. Thermocouples placed in a stack above the upper end of the specimen furnish the necessary indication of the rate at which the flames would supply heat to unignited material under normal burning conditions.

APPARATUS

Figure 1 presents a view of the experimental apparatus developed for performance of this radiant panel flame spread test. The essential components shown here include the radiant panel with its associated air and gas supply system, the specimen holder and framework for its support, the stack with thermocouples, the smoke sampling tube with its gas metering system, and the radiation pyrometer. Not shown are a manually operated potentiometer, a recording potentiometer, and a decimal minute timer. A brief description will be given of each of these components.

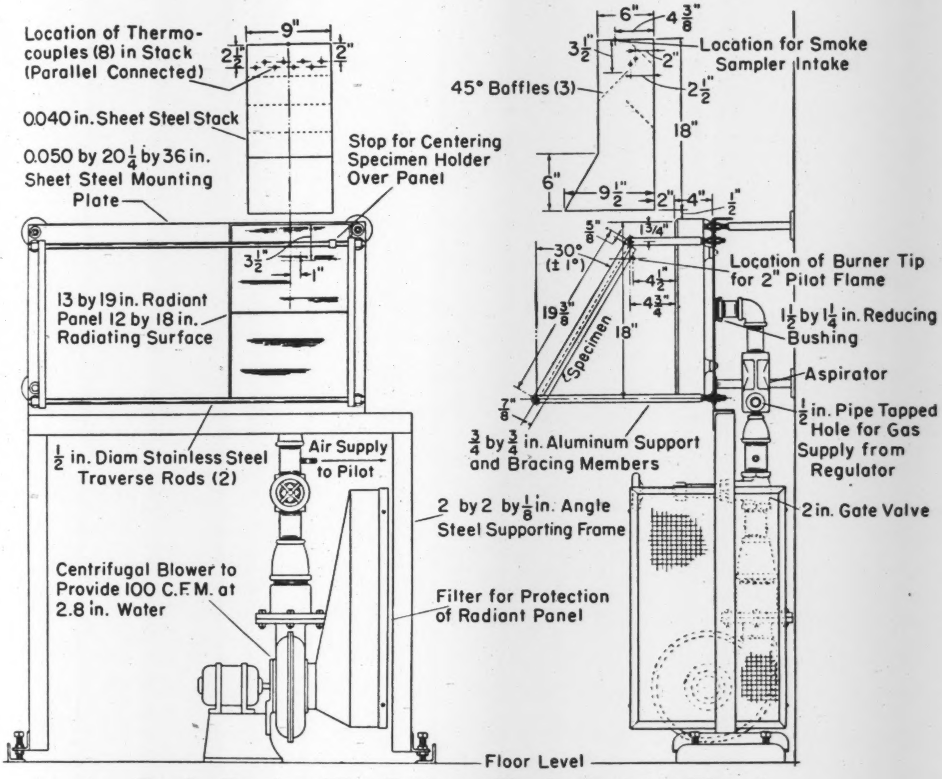
Location of Thermocouples (8) in Stack (Parallel Connected)
9"
$2\frac{1}{2}$"
$\frac{1}{2}$"
0.040 in. Sheet Steel Stack
0.050 by $20\frac{1}{4}$ by 36 in. Sheet Steel Mounting Plate
Stop for Centering Specimen Holder Over Panel
$3\frac{1}{2}$"
1"
13 by 19 in. Radiant Panel 12 by 18 in. Radiating Surface
$\frac{1}{2}$ in. Diam Stainless Steel Traverse Rods (2)
Air Supply to Pilot
Centrifugal Blower to Provide 100 C.F.M. at 2.8 in. Water
Filter for Protection of Radiant Panel
2 by 2 by $\frac{1}{8}$ in. Angle Steel Supporting Frame
6"
$4\frac{3}{8}$"
$3\frac{1}{2}$"
2"
Location for Smoke Sampler Intake
45° Baffles (3)
$2\frac{1}{2}$"
18"
6"
$9\frac{1}{2}$"
2"
4"
$\frac{1}{2}$"
$\frac{5}{8}$"
$1\frac{3}{4}$"
30° (± 1°)
$4\frac{1}{2}$"
$4\frac{3}{4}$"
$19\frac{3}{8}$"
18"
Specimen
$\frac{7}{8}$"
Location of Burner Tip for 2" Pilot Flame
$1\frac{1}{2}$ by $1\frac{1}{4}$ in. Reducing Bushing
Aspirator
$\frac{1}{2}$ in. Pipe Tapped Hole for Gas Supply from Regulator
$\frac{3}{4}$ by $\frac{3}{4}$ in. Aluminum Support and Bracing Members
2 in. Gate Valve
Floor Level

FIG. 3.—Drawing of Test Equipment Showing Important Dimensions.

Radiation Panel:

The radiant panel consisted of a cast iron frame enclosing porous refractory material, the assembly being imported from England.[8] The panel was mounted in a vertical plane. It was fed from the rear with a premixed gas-air supply, combustion of which was nearly completed within its pores. After warm-up, the only visible flames from the panel surface were less than $\frac{1}{8}$ in. in length. The panel approaches its equilibrium temperature in about one half hour after ignition. This particular panel was selected as being the only one available at the time which could operate at temperatures as high as 1500 F. It is quite possible that other types of burners or even electrical heaters would prove equally satisfactory. Electrically heated panels were not considered because of the power consumption which amounted to about 24 kw based on the total heating value of the gas supplied for combustion.

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[8] The appendix includes a list of components used and in cases of special requirements or limited availability suggests a possible source of supply. It is possible however that other sources of supply while unknown to the writers do exist.

Air was supplied to the panel by means of a small centrifugal blower fitted with an air filter to prevent dust from obstructing the panel pores. Gas was premixed with air in a Venturi mixer to which it was supplied at approximately atmospheric pressure.

Specimen Holder:

Figure 2 presents an assembly drawing of the specimen holder used for support of the 6 by 18-in. specimens consisting of a simple device formed of sheet steel. Although low-carbon steel was used in the initial holders, it would probably be preferable to use a heat resisting chromium steel in their fabrication. Figure 3 presents general arrangement drawings of the radiant panel and support frame necessary for proper positioning of the specimen. An attempt has been made to show only those dimensions which should be maintained in reproduction of the equipment. The angle of the specimen and its position with respect to the panel were found to be critical and have been selected as a result of a series of tests.

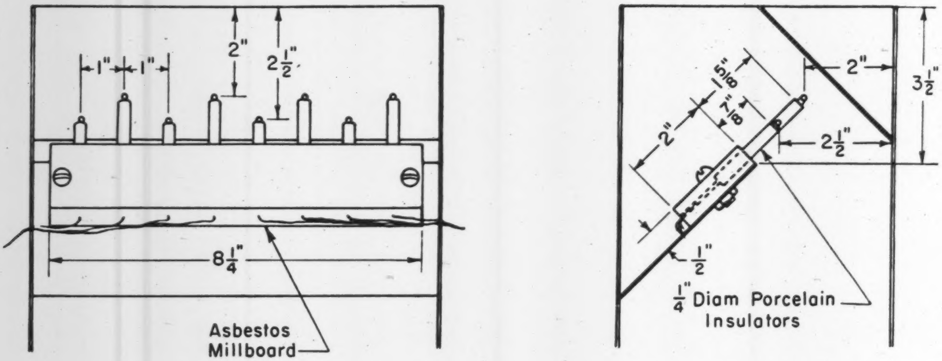

FIG. 4. Detail of Thermocouple Mounting Arrangement.

These drawings also show the position of the tip of the pilot burner tube which was used to force ignition, when this was possible, at the top of the specimen. This burner consists of a short length of $\frac{3}{16}$ in. outside diameter by $\frac{1}{8}$ in. inside diameter stainless steel tubing, the portion of which exposed to radiant energy from the panel was protected by an outer porcelain tube $\frac{9}{32}$ in. in outside diameter by $\frac{13}{64}$ in. in inside diameter. It has been found necessary to replace this burner occasionally because of oxidation and residue deposits which collect on it. To cut down on the frequency of such replacement, the pilot has been arranged to swing out of position when not in use.

The pilot was fed with acetylene premixed with air supplied from the radiant panel air supply blower. Use of an aspirating type fitting might prove to be a more desirable method of supplying the necessary air.

Stack and Thermocouples:

Figure 3 shows the dimensions and position of the stack with respect to the specimen and radiant panel, as well as the location of the eight thermocouple junctions. These junctions were formed by fusing the end of a twisted pair of Chromel and Alumel wires of 0.020 in.

diameter. The junctions were supported in position by means of porcelain insulators as shown in Fig. 4. The group of eight thermocouples were of equal resistance and were connected in parallel, the output being recorded by an automatic potentiometer recorder.

Smoke Sampling Device:

The smoke sampling device, Fig. 5, consisted simply of a means for support of a single layer of glass fiber filter paper at a given position above the stack. An aspirator and rotameter were used to maintain a constant air flow velocity of 40 ft per min (20.4 cm per sec) at the face of the filter. The method of collecting and weighing the smoke deposit was used because of the usual difficulties experienced with sampling techniques and condensation problems experienced with optical methods of smoke measurement.

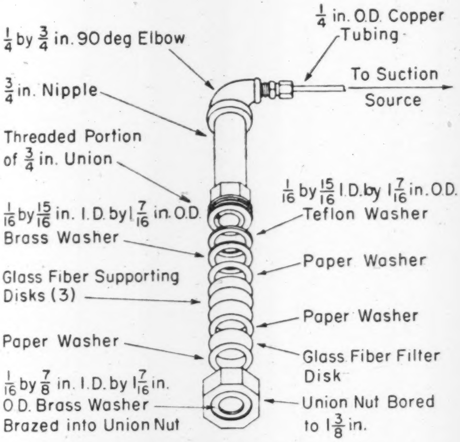
$\frac{1}{4}$ by $\frac{3}{4}$ in. 90 deg Elbow
$\frac{1}{4}$ in. O.D. Copper Tubing
To Suction Source
$\frac{3}{4}$ in. Nipple
Threaded Portion of $\frac{3}{4}$ in. Union
$\frac{1}{16}$ by $\frac{15}{16}$ I.D. by $1\frac{7}{16}$ in. O.D. Teflon Washer
$\frac{1}{16}$ by $\frac{15}{16}$ in. I. D. by $1\frac{7}{16}$ in. O.D. Brass Washer
Paper Washer
Glass Fiber Supporting Disks (3)
Paper Washer
Paper Washer
Glass Fiber Filter Disk
$\frac{1}{16}$ by $\frac{7}{8}$ in. I. D. by $1\frac{7}{16}$ in. O.D. Brass Washer Brazed into Union Nut
Union Nut Bored to $1\frac{3}{8}$ in.

FIG. 5.—Smoke Sampling Tube and Parts.

Associated Equipment:

A radiation pyrometer was used for standardization of the thermal output of the panel. The electrical output of this device was monitored by means of a portable manually operated potentiometer. An automatic recording potentiometer was used for recording stack thermocouple temperature variations. The instrument used had a range of 32 to 932 F (0 to 500 C). A small timer calibrated in minutes and decimal fractions to hundredths was used for recording the time of occurrence of events during the tests.

TEST PROCEDURE

The procedure developed for performance of the flame spread test may be outlined as follows:

(*a*) Gas passing through the radiant panel was ignited and the unit allowed to heat for a one half hour period. At the end of this time a check was made of the radiant output by means of the radiation pyrometer placed in such a manner as to view a central panel area of about 10 in. diameter. If necessary, adjustment was made in the rate of gas supply to maintain the radiant output at that which would be obtained from a black body of the same dimensions operating at a temperature of 1238 F (670 C).

(*b*) The recording potentiometer for measurement of stack thermocouple temperature rise was turned on and if necessary the thermocouples were cleaned by use of a brush.

(*c*) A new filter disk was placed in the smoke sampling tube and the air flow rate adjusted.

(*d*) The pilot was ignited, adjusted to provide a flame 2 or 3 in. in length, and placed in position in front of the radiant panel.

(*e*) A 6 by 18 in. specimen previously conditioned to equilibrium with an ambient of 75 ± 5 F and 50 ± 5 per cent relative humidity was either (1) mounted in a cool specimen holder in the conditioning room and brought to the test equipment in a vapor barrier jacket, or (2) wrapped in a vapor barrier jacket, brought to the test equipment, and quickly inserted in a cool specimen holder. In either case, all but the thickest specimens were backed with $\frac{1}{2}$ in. thick asbestos millboard of 60 lb per cu ft density.

(*f*) The smoke sampling tube was placed in position above the stack.

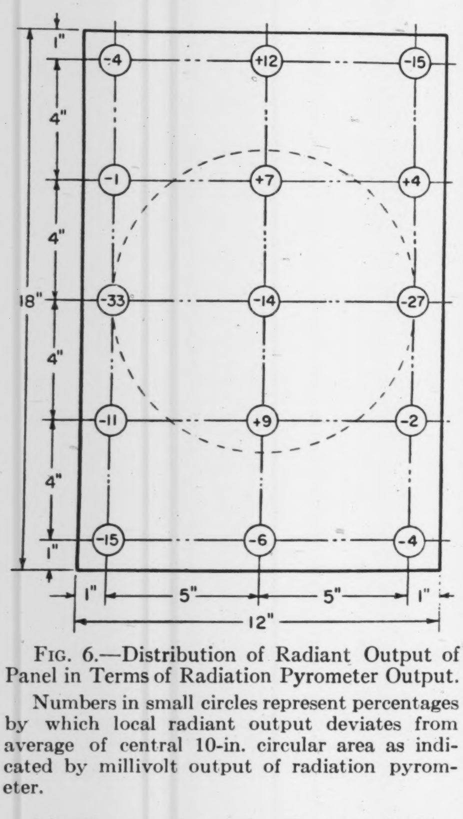

FIG. 6.—Distribution of Radiant Output of Panel in Terms of Radiation Pyrometer Output.

Numbers in small circles represent percentages by which local radiant output deviates from average of central 10-in. circular area as indicated by millivolt output of radiation pyrometer.

(*g*) The specimen in the test holder was placed in position on the radiant panel frame and the electrical timer simultaneously started. The exposure of the specimen took place as promptly as possible after bringing it to the test area. In most cases this was accomplished within 3 min. In no case were conditioned specimens allowed to stand in the test room for more than 5 min prior to test.

(*h*) Observations were continuously made of the development and spread of flaming on the specimen. Records were kept of the time of arrival of the flame front at points 2, 3, 6, 9, 12, 15, and 18 in. from the upper edge of the specimen. Notes were made of any other specimen behavior characteristics which appeared of interest.

(*i*) The test exposure was considered as completed when either (1) the flame front had progressed the full length of the specimen, or (2) the test exposure period of 15 min had elapsed. The specimen holder was then removed from the supporting frame and the specimen discarded.

(*j*) Measurements of the increase in weight of the smoke sampling filter paper disk and the maximum temperature rise of the stack thermocouples were recorded.

(*k*) The flame spread index, I_s, of a sample was calculated as the product of the flame spread factor, F_s, and the heat evolution factor, Q, thus:

$$I_s = F_s Q$$

where:

$$F_s = \left(1 + \frac{1}{t_3} + \frac{1}{t_6 - t_3} + \frac{1}{t_9 - t_6} + \frac{1}{t_{12} - t_9} + \frac{1}{t_{15} - t_{12}}\right)$$

here:

$t_3 \ldots\ldots t_{15}$ correspond to the times in minutes from specimen exposure until arrival of the flame front at a position of 3 ..... 15 in., respectively, along the length of the specimen.

$$Q = 0.1\Delta\theta/\beta.$$

here:

0.1 is a constant arbitrarily chosen to yield a flame spread index of approximately 100 for red oak, $\Delta\theta$ is the observed maximum stack thermocouple temperature rise in degree Fahrenheit over that observed with an asbestos cement board specimen, and β is the maximum stack thermocouple temperature rise for unit heat input rate to the calibration burner, deg Fahr min per Btu.

CALIBRATION PROCEDURES

The calibration procedures used during development of the test method were of two different types. First, certain procedures were found to be necessary for standardization of the test method to permit reproducibility of test results. These will be designated as "standardization procedures." Secondly, a series of tests was performed in an effort to determine the sensitivity of the test method to small changes in orientation or assembly of the test equipment. These studies are described under "procedural variations."

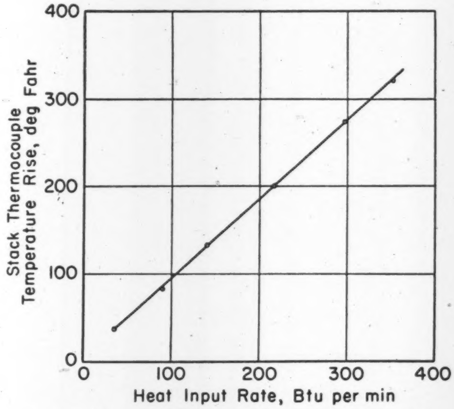

FIG. 7.—Typical Stack Thermocouple Calibration Curve.

Standardization Procedures:

The radiant output of the panel was monitored continuously between tests by means of the radiation pyrometer. Adjustments in gas flow rate were frequently and easily made. As previously mentioned, this radiant output rate, per unit area, was maintained at that of a black body radiating at a temperature of 1238 F (670 C). Figure 6 shows the variation of radiant output in terms of radiation pyrometer output voltage for the panel used. In making the survey, the pyrometer was located in a position to view a panel area of about 1-in. diameter.

The smoke sampling tube and associated connections were checked periodically by use of an impermeable diaphragm in place of the filter disk. Any flow that could be measured with the inlet thus blocked was a sure indication of leaks. On several occasions leaks were detected. Such leaks are serious, as the filter resistance to gas flow is not negligible and leaks would therefore result in low recorded smoke weight.

Periodic checks were made of the temperature rise produced by placing an asbestos-cement board specimen in position while both the radiant panel and pilot burner were in operation.

No test method was devised to check for objectional deposits of combustion products on the thermocouples other than periodic inspection. For most cellulosic materials tested, it was found that inspection and brush cleaning of the thermocouples was desirable at least once every two weeks of operation for eight hours a day. When testing hardboard specimens as well as plastics, the smoke production rate required much more frequent attention to this detail.

Calibration of the stack thermocouples was performed by means of a diffusion flame from a multi-ported burner. This burner (ten 0.070 in. in diameter holes spaced $\frac{5}{8}$ in. on centers in a $\frac{1}{4}$-in. standard pipe) was placed in a horizontal position 1 in. below the upper edge of an asbestos-cement board specimen during normal panel operation. From observations of the maximum stack thermocouple temperature rise as a function of heat supply rate to the test burner the value of β in the flame spread index formula was measured. For the equipment used here, β was found to be equal to 0.90 deg Fahr min per Btu. Figure 7 presents a plot of data obtained. There was no indication that this measurement required frequent checking.

One other calibration was performed to provide an indication of the heat input rates at the specimen surface. This involved radiometer measurements at various positions along the specimen length. The measurement was made by the use of a small copper disk radiometer fitted in one opening at a time of a series of apertures on an asbestos millboard specimen. The radiant panel was in operation, but the pilot was not burning during these measurements. Figure 8 presents a calibration curve obtained in this way. It should be noted that the energy flux measured and indicated here was the sum of both the radiant flux and that arising from convection effects.

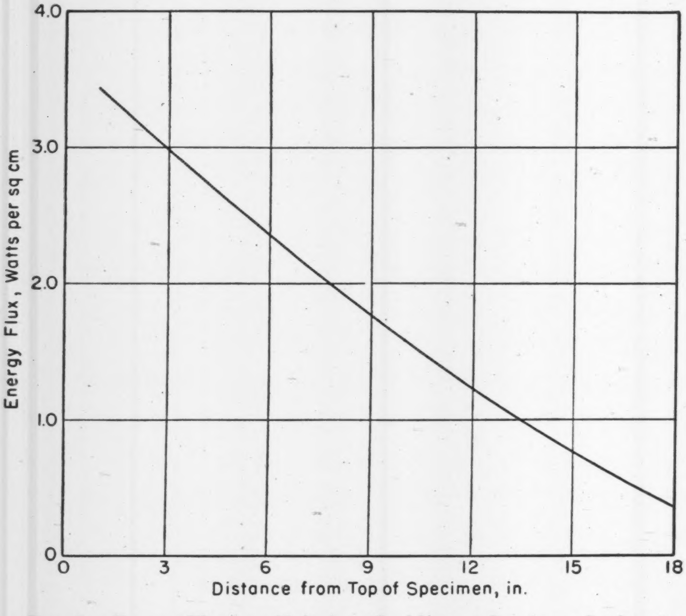

FIG. 8.—Energy Flux per Unit Area Incident at Specimen Surface.

Procedural Variations:

A number of tests were performed for the purpose of determining the extent to which design features of the equipment were critical.

The position of the specimen relative to the radiating face of the panel was found to be critical. This position determines the energy flux at the specimen surface. While measurements were not made on the effects of changes in positioning, it appears likely that the dimensions indicated in Fig. 3 should be maintained to a tolerance of less than $\frac{1}{8}$ in.

The technique used for calibration of stack thermocouples was selected to reduce the effects of slight differences in design or positioning of the parts of the apparatus. Figure 9 presents data on the effect of stack position on thermocouple temperature rise for both a typical specimen (fiberboard No. 8) and the calibration burner. It is evident from the plot of the temperature rise *versus* horizontal clearance for this specimen that exact maintenance of the 2-in. spacing is not required during test. Although this behavior of fiberboard is typical of all materials tested, the same insensitivity is not observed when the calibration burner is used as a heat source. This probably results from differences which exist in the manner of heat release in the two cases. Because of the effect of the spacing on the value of β, it is important to maintain the correct stack position during calibration. Changes in vertical position of the stack and their effect on thermocouple temperature rise measured during test of specimens were not investigated. It is expected however that the data would be quite similar to that shown in Fig. 9 for the calibration burner.

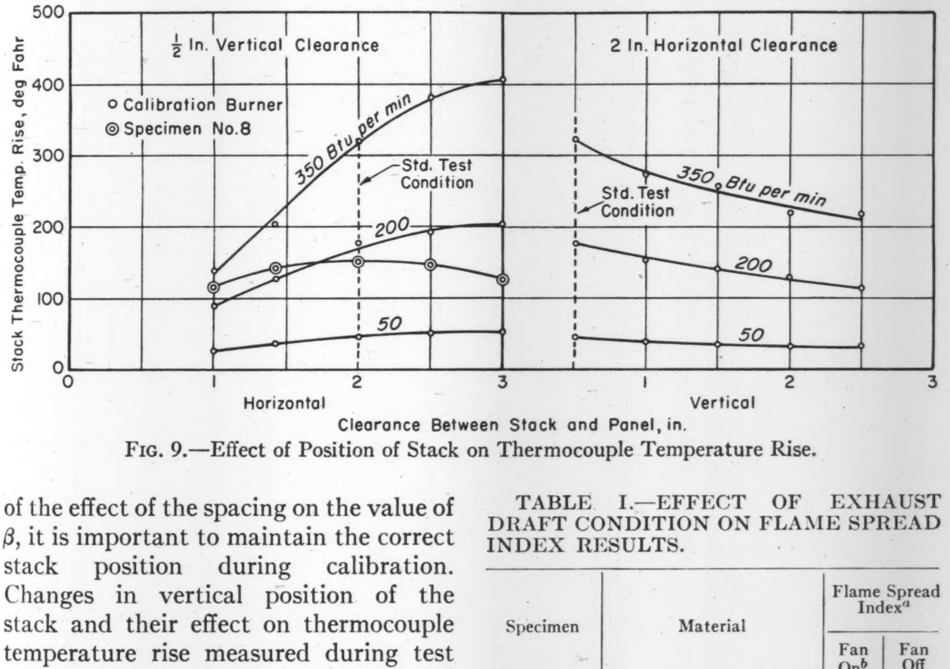

FIG. 9.—Effect of Position of Stack on Thermocouple Temperature Rise.

The importance of position of the pilot flame was studied. It was found that the size of this flame and its position with respect to the face of the specimen were not critical as long as the flame remained in contact with or at most not more than ½ in. from the surface of the specimen.

Conditioning of the specimen prior to test was important as evidenced by data obtained for fiberboard specimen No. 8. Flame spread index values of 353, 236, and 156 were measured for specimens conditioned to have 1, 7, and 12 per cent of moisture by weight, respectively. These moisture equilibrium contents correspond to widely different ambient relative humidity conditions. Because of this and the shape of the

TABLE I.—EFFECT OF EXHAUST DRAFT CONDITION ON FLAME SPREAD INDEX RESULTS.

Specimen	Material	Flame Spread Index[a]	
		Fan On[b]	Fan Off
No. 28.....	Fiberboard A	278	281
No. 23.....	Hardboard Common M	114	116
No. 21.....	Fiberboard C	61	85
No. 26.....	Gypsum Board G	4.7	4.6

[a] Average flame spread index for two specimens. All tests run on same day.

[b] Differences between these test results and the data shown in Table II result from the limited number of samples tested and from variations between samples.

TABLE II.—RADIANT PANEL FLAME SPREAD DATA.

Specimen	Materia				Flame Spread Data					Classification by Other Methods			Smoke Data		
	Name	Nominal Thickness, in.	Bulk Density, lb per cu ft	Moisture Content, per cent[a]	Number of Tests	Flame Spread Factor, F_s	Heat Evolution Factor, Q	Flame Spread Index, I_s	Coefficient of Variation, v, per cent	Underwriters' Laboratory Flame Spread Factor	SS-A-118b Classification	Flashover Time,[d] min	Deposit, mg	Based on Red Oak	Underwriters' Laboratory Smoke Factor
No. 17	Fiberboard perforated tile, finish removed	½	15.7	5.4	5	23.7	14.1	336	14.7				0.1	30	
No. 28	Fiberboard A (FPL)[c]	½	20.0	8.0	6	15.3	18.2	278	5.1	285		5.00	0.4	130	357
No. 8	Fiberboard J unfinished	½	18.0	7.0	9	18.2	12.8	236	5.0				0.2	70	
No. 30	Plywood exterior fir unfinished	¼	42.3	6.8	4	8.5	17.0	143	32.2		D		0.3	100	
No. 18	Hardboard Common H	7/32	59.8	3.8	5	4.5	30.2	136	12.4				4.1	1400	
No. 23	Hardboard Common M (FPL)	¼	63.5	3.8	6	4.1	29.7	121	6.9	113	D	6.15	5.2	1700	1900
No. 16	Fiberboard perforated tile	½	16.7	5.8	5	10.0	11.6	116	13.6				0.3	100	
No. 13	Fiberboard J + paint A, 250 sq ft per gal	½	18.3	6.5	5	8.4	12.2	107	44.0				0.4	130	
No. 24	Red oak (FPL)	¾	40.0	6.5	5	7.0	14.1	99	10.9	100	D		0.3	100	100
No. 21	Fiberboard C (FPL)	½	19.7	6.0	5	10.3	9.0	94	18.3	77	D		0.2	70	94
No. 9	Fiberboard J factory finish	½	20.2	6.6	5	9.9	8.4	83	15.9				0.2	70	
No. 12	Fiberboard J + paint B, 250 sq ft per gal	½	17.0	6.2	5	6.0	9.8	59	17.2		D		0.6	200	
No. 34	Plastic C	⅛	81.5		5	4.9	11.5	56	8.4	130			9.7	3200	752
No. 33	Plastic B	⅛	98.0		5	3.7	8.4	30	19.5	60			5.7	1900	368
No. 11	Fiberboard J + paint B, 125 sq ft per gal	½	17.8	6.9	5	4.1	6.4	27	26.0			10.35	0.9	300	
No. 26	Gypsum board G (FPL)	⅜	50.5		5	4.2	3.6	14.3	44.5	10	C		0.0	0	0
No. 3	Gypsum board K	⅜	47.4		5	4.4	3.1	13.3	29.9				0.1	30	
No. 6	Mineral base tile	¾	19.3	3.4	5	7.8	1.5	10.5	60.0	10[b]			0.0	0	
No. 25	Gypsum board L	½	51.2		4	3.0	2.3	7.9	105.0	12.8		16.36	0.1	30	17
No. 22	Fiberboard I fire retardant, finish (FPL)	½	19.0	6.4	5	1.8	2.6	4.5	53.8	14	D		0.5	170	14
No. 32	Plastic A	⅛	89.4		5	1.0	4.0	4.0	42.3	20			3.3	1100	556
No. 14	Fiberboard J + mineral spray	½	21.9	7.7	5	1.0	3.4	3.4	28.9				0.2	70	
No. 7	Glass fiber batt	1	4.3	1.2	5	1.0	1.8	1.8	60.5				0.2	70	
No. 2	Cellulose mineral board	⅞	47.8	7.5	5	1.0	1.3	1.3	48.6	9–10[b]		16.45	0.1	30	0[b]
No. 5	Glass fiber tile	⅜	10.3	0.6	5	1.0	1.2	1.2	77.7	10–15[b]			0.3	100	
No. 15	Fiberboard J + ⅛ in. mineral surface	⅝	42.2	7.5	5	1.0	1.2	1.2	50.7		C		0.2	70	
No. 31	Plywood exterior fir + aluminum foil	¼	41.6	6.3	3	1.0	0.9	0.9	65.7		D		0.1	30	
No. 4	Glass fiberboard	2	11.0	0.4	5	1.0	0.6	0.6	37.3		B		0.2	70	
No. 1	Asbestos cement board	3/16	117.0	0.6	5	1.0	0.0	0.0		0	A	17.10	0.0	0	

[a] Based on weight after drying at 221 F (105 C).

[b] Tests on same materials but samples from other batches.

[c] Designates materials received from Forest Products Laboratory.

[d] Results of British Tests on somewhat similar materials (**5**).

moisture equilibrium *versus* relative humidity curve for materials such as these, the effect of small changes from the standard conditioning temperature and humidity would not be expected to result in flame spread index changes proportional to those indicated above.

A study was made on the effect of variations in draft conditions on the measured flame spread index. Most of the test results reported in this paper were obtained while the furnace was operating under a 2 by 4-ft hood, the exhaust blower of which was in operation. The air flow rate was over 600 cu ft per min through this hood. Table I presents average flame spread index data from tests of two specimens of each of four different materials. These tests were performed both with and without operation of the exhaust fan. It will be observed that when proper calibration has been made and used in flame spread index calculation, the test results are very similar for the two test conditions. Even when the results are not corrected for change of thermocouple calibration, the flame spread indices became 256, 105, 77, and 4.2—a change of only about 10 per cent.

The apparent contradiction to this generalization, presented by the data for specimen No. 21, is believed to have been caused by nonuniformity of the specimens and the limited number of samples tested.

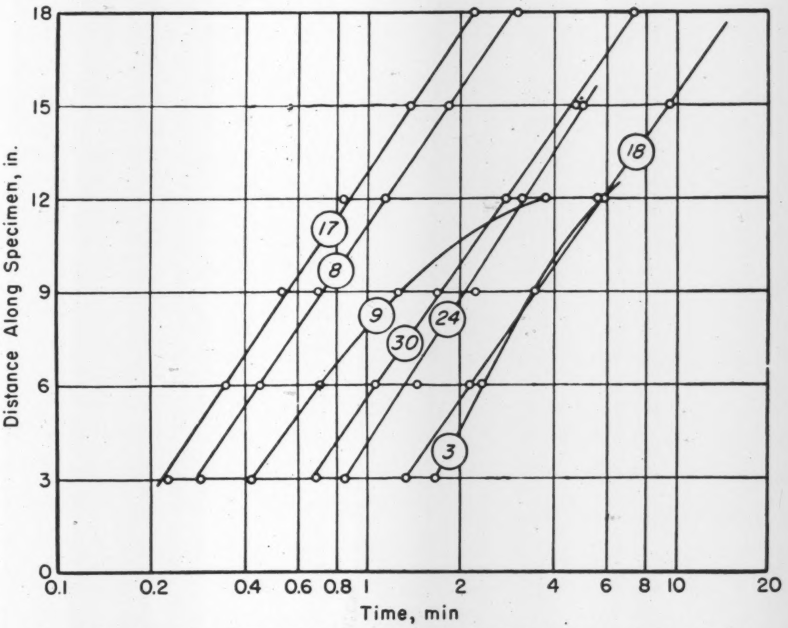

FIG. 10.—Progress of Flame Along Specimen Length for Several Materials. Numbers correspond with specimen number in Table II.

DISCUSSION

The test method has been used to measure the flame spread properties of more than 60 different materials. Data on the behavior of 29 of these are included in Table II. The materials are listed in order of decreasing flame spread index. The constant in the flame spread index relation was arbitrarily chosen to yield a value of approximately 100 for red oak. Included in this table are data on all materials thus far studied for which Underwriter tunnel test data were available as a result of tests performed on samples from the same material batch. The flame front progression along the surfaces of various specimens is shown in Fig. 10. The numbers on the curves correspond to specimens described in Table II.

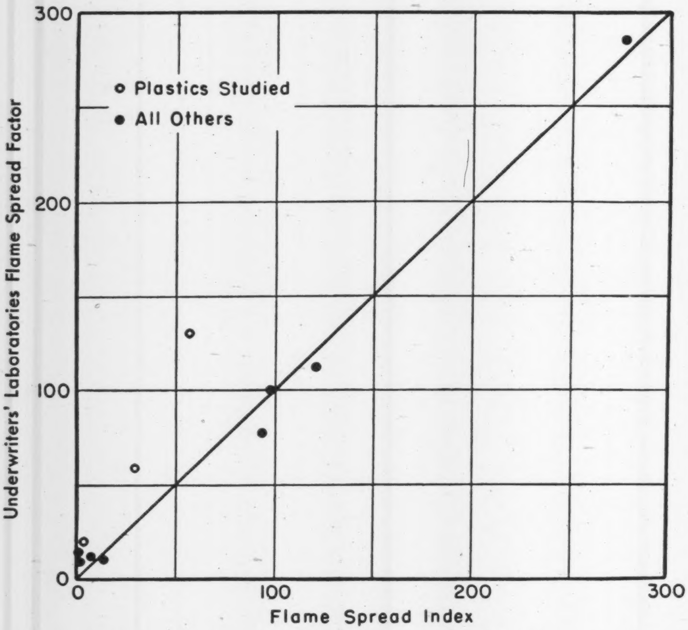

FIG. 11.—Comparative Data from Two Different Flame-Spread Test Methods.

Brief study of the data presented indicates that the method selected for calculation of the flame spread index results in a numerical grading of specimens which is in general accord with experience. Furthermore, for flame spread index data above about seven and with the exception of data for plastic materials, there appears to be a fair degree of correlation between the classification of materials by both the radiant panel and tunnel test methods (Fig. 11). The flame spread index values resulting from tests of the three polyester plastic materials were only about one half the classification figures assigned on the basis of tunnel tests. Specimen No. 32 (plastic A) did not exhibit sustained flaming and only periodic flashes were observed in the gases leaving the specimen. A number of the materials were tested by the horizontal panel test described in Federal Specification SS-A-118b with the results given in Table II. It is evident that this method of test does not measure the same surface flame spread property as measured by the radiant panel or tunnel test methods.

Experimental results of tests by the British Spread-of-Flame Test are not available on the materials in Table II. However, Hird and Fischl (5) present the results of a study of flash-over times observed during a series of tests with model rooms which were finished with a wide variety of materials some of them apparently similar to those tested here.

These latter have been listed in Table II. It will be observed that the order in which the materials are classified is similar to that achieved with the radiant panel test method. The model test method however appears to show little difference in unfinished fiberboards whereas the method of interpretation of the radiant panel and tunnel results shows flame spread indices varying from 121 to 336.

The flaming behavior of the specimens might be selected as an indication of low, intermediate and high flame spread characteristics. Thus specimens with a flame spread index of less than four, rarely exhibited sustained surface flaming. In the index range of 4 to 140 sustained flaming usually occurred only over a portion of the specimen. For flame spread indices above 140 flaming occurred over the full specimen length during the 15 min test period.

TABLE III.—REPRODUCIBILITY OF TEST DATA.

Specimen	Material	Tests on One Day		Tests of a Period of Month	
		Number of Specimens	Coefficient of Variation, v, per cent	Number of Specimens	Coefficient of Variation, v, per cent
No. 24.....	Red Oak	5	10.9		
No. 19.....	Red Oak[a]			19	18.5
No. 23.....	Hardboard	6	6.9		
No. 28.....	Fiberboard "A"			8	8.8
No. 8.....	Fiberboard "J"	8	5.1	9	5.0

[a] Includes data on samples covering a density range of 30.7 to 46.9 lb per cu ft.

The flame spread characteristics measured are only those of the exposed surface of the specimen. Evidence of this is provided by Specimens 8, 13, 9, 12, 11, 14, and 15 which all incorporate essentially the same ½-in. fiberboard backing and yet with the varying types of surface treatment exhibit flame spread index values ranging from 244 down to 1.2. This should provide ample evidence that consideration should be given to the maintenance of an unbroken protective surface on the material when used as finish material. In cases where materials of this type are used for application directly to studs or over other enclosed open spaces it appears highly desirable that both surfaces be treated in the same fashion. In cases such as this, Underwriters' Laboratories handles the problem by specifying that the classification assigned is only applicable when the finish is applied over a solid backing.

Some appreciation of the reproducibility of the test method may be derived from the data presented in Table III. Here, v, the coefficient of variation (the standard deviation expressed as a percentage of the mean value of the flame spread index) is shown for four different materials. The value of v has been computed from the individual values of the flame spread index for the replicate tests performed. It will be noticed that v for specimen No. 8 is very nearly of the same magnitude for a series of tests performed on a single day as compared to the value for tests run over a period of time, in this case 120 days. Thus the major portion of the variability of test data appears to be a result of variations, either between specimens, or in the test method itself within a single day. Over the flame spread index range of 121 to 278 represented by Specimens Nos. 8, 23, and 28, the coefficient of variation is within the range 5.0 to 8.8 per cent. It thus appears to be relatively independent of the flame spread index for such materials. The value of $v = 18.5$ per cent for red oak specimens which were selected to cover a range of densities appears to indicate the increase in variability of the samples themselves.

The data shown for both calibration

burner and fiberboard specimen in Fig. 9 make it quite clear that the calibration method used should not be considered as providing an absolute calibration of heat output rates of materials. The refinements necessary to achieve this objective were considered inappropriate for the application intended.

Smoke measurements are also reported in Table II. These are reported as milligrams of smoke deposit for the volume flow rate maintained during the period of test. The results are also presented on a relative basis using a value of 100 for red oak which is the standard used by Underwriters' Laboratories. There appears to be a general correlation although big differences are observed in several instances. For materials exhibiting low flame index data, it appears that higher observed smoke release rates are obtained with the radiant panel test method than with the tunnel test method.

The test method described appears to present a simple reproducible method of measuring the flame spread behavior of building finish materials. The method of presentation of test results was arbitrarily selected to yield classification numbers somewhat similar to those reported by Underwriters' Laboratories on the basis of tunnel tests. Because of the lack of quantitative data on, and the intangible nature of, the flammable "hazard" of materials, it is not at present possible to define the exact relation which exists between flame spread index and the actual hazard of the material.

CONCLUSIONS

The following conclusions seem justified on the basis of the work reported:

1. A flame spread test method has been devised for measuring the flammability characteristics of interior finish materials.

2. The test method involves the use of comparatively inexpensive and compact test equipment and requires test specimens of but 6 by 18 in. size.

3. A method for expression of test results has been devised which appears to classify materials in an order similar to that indicated as desirable from experience with such finish materials.

4. With some exceptions there appears to be a fair correlation between the flame spread results as obtained by the radiant panel and the tunnel test method over the flame spread index range of 10 to 280.

APPENDIX

List of components used with a possible source of supply for those of special requirements or limited availability.

1. *Radiant panel*—12 by 18 in. type 1 surface combustor, manufactured by Radiant Heating Ltd., Barnsbury Park, London N. 1, England.

2. *Radiation pyrometer*—Suitable for viewing circular area of ten inch diameter at a range of about 4 ft and calibrated over black body temperature range of 0 to 1900 F.

3. *Glass fiber paper*—Type 1106BH All-Glass Filter Media, distributed by Mine Safety Appliances Co., 201 N. Braddock Ave., Pittsburgh 8, Pa.

4. *Potentiometer recorder*—Range: 0 to 1000 F.

5. *Portable potentiometer*—Provided with millivolt range corresponding to 0 to 1900 F for use with radiation pyrometer.

6. *Flowmeter*—Capacity: 7000 ml air per min.

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(3) H. D. Bruce, "Experimental Dwelling Room Fires," United States Dept. of Agriculture, Forest Service, Forest Products, Report No. D1941, June, 1953.

(4) A. J. Steiner, "Fire Hazard Classification of Building Materials," *Bulletin of Research*, No. 32, Sept., 1944, Underwriters' Labs., Chicago.

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ADDENDUM

Since preparing this paper, the following modifications or precautions in use of this test method have appeared desirable:

1. Translucent materials were backed with a sheet of highly reflective aluminum foil. This was done for the purpose of standardizing the testing procedure for such materials.

2. No attempt has been made to measure flame spread behavior of materials having surface corrugations of greater than ½ in. depth.

3. While statements in the text relative to the lack of sensitivity of the test method to draft conditions above the stack appear justified as far as flame spread index is concerned this does not appear to hold for the smoke measurement. It seems desirable to have sufficient draft to produce average gas velocities at the outlet of the stack of 100 ft per min with the panel cold. This corresponds with about 250 ft per min with the panel in operation. Any hood used to produce these draft conditions should clear the top and sides of the stack by a minimum of 10 and 7½ in., respectively.

4. The glass filter disks used appear to have a small but significant equilibrium moisture content. This should be accounted for in reporting smoke measurements.

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON ION-EXCHANGE AND CHROMATOGRAPHY IN ANALYTICAL CHEMISTRY

A series of annual symposia is planned on timely subjects of importance to ASTM members concerned with analysis of materials, to acquaint them with new methods, techniques, etc., which are actually or potentially capable of use in standard methods of analysis. This Symposium on Ion-Exchange and Chromatography in Analytical Chemistry is the first in the series, and it is intended to present a picture of the materials available, or under development, for ion-exchange and chromatography, the basic theory of their use, and examples of their application for separation and analysis of materials, primary metals, though it includes valuable data concerning other materials.

The papers and discussions of this Symposium were presented at the fourth session of the Fifty-ninth Annual Meeting of the American Society for Testing Materials, held in Atlantic City, N. J., June 18, 1956. It was sponsored by ASTM Committee E-3 on Chemical Analysis of Metals, and Division G of this Committee constituted the Symposium Committee with H. F. Beeghly of Jones & Laughlin Corp., Pittsburgh, Pa. acting as Symposium Chairman.

The papers included in the Symposium are:

The Use of Ion-Exchange Resins in Analytical Chemistry—Robert Kunin
The Use of Ion-Exchange Techniques in Analytical Chemistry and Radiochemistry—J. E. Hudgens
Anion-Exchange in Metal Separations—Kurt Kraus
Ion-Exchange in the Analysis of Metals—John L. Hague

These papers with discussions were issued as *ASTM Special Technical Publication No. 195*, entitled "Symposium on Ion-Exchange and Chromatography in Analytical Chemistry."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON pH MEASUREMENT

This Symposium on pH Measurement was sponsored by Subcommittee 22 on Hydrogen Ion Determinations of Committee E-1 on Methods of Testing and presented at the Sixth and Tenth Sessions of the Fifty-ninth Annual Meeting of the American Society for Testing Materials held in Atlantic City, N. J., June 19, 1956. The plans and arrangements for this Symposium were made by a committee composed of Roger G. Bates, National Bureau of Standards, Washington, D. C., Clinton Grove Central Commercial Co., Northfield, Ill., G. A. Perley, Leeds & Northrup, Philadelphia, Pa., and E. B. Ashcraft, chairman of Subcommittee 22, Westinghouse Research Laboratories, Westinghouse Electric Corp., East Pittsburgh, Pa. Mr. Bates acted as chairman of the Sixth Session at the Meeting and Robert Burns, National Research Council, Washington, D. C. was chairman of the Tenth Session.

In the ten years that have elapsed since the first Symposium on pH Measurement was held (STP 73, 1946), significant developments in pH instrumentation and technology have been made. Agreement on standard pH methods is gradually being achieved as a single conventional standard scale comes slowly into general use throughout the world. Techniques have been devised for the accurate measurement of pH under specialized conditions of unusual difficulty, and much thought has been given to the precise role of pH in reactions of interest to the analytical and physical chemist. The increased laboratory and industrial importance of nonaqueous and partially aqueous solvents has stimulated the search for functions that will give a useful indication of the level of acidity and basicity in these media. It is the purpose of this second Symposium on pH Measurement to consider these new developments and to clarify the problems they have created.

The Symposium includes the following papers:

Foreword—E. B. Ashcraft
Meaning and Standardization of pH Measurements—Roger G. Bates
Performance Studies of Reference Electrodes and Their Components at High Temperatures and Pressures—John E. Leonard
Modern Developments in pH Instrumentation—W. R. Clark and G. A. Perley
Problems in Measurement of the pH Measurement in Analytical Chemistry—Julius Sendroy, Jr.
Quantitative Applications of pH of Blood and Other Biological Fluids—Henry Freiser
Theoretical and Practical Problems in the Measurement of Acidity in Nonaqueous Media—Martin Kilpatrick
Indicator Acidity Functions for Nonaqueous and Mixed Solvents—M. A. Paul and F. A. Long

These papers with discussion were issued as *ASTM Special Technical Publication No. 190* entitled "Symposium on pH Measurement."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON TENSION TESTING OF NON-METALLIC MATERIALS

Among the non-metallic materials there is such a diversity of structures and properties that a variety of methods for measuring tensile properties has evolved. New technical tools, new materials, and new understanding of the structure of materials has brought a more meaningful interpretation of results. At the same time these new concepts have contributed greatly to the terminology and nomenclature of tension testing.

The papers and discussions in this Symposium on Tension Testing of Non-Metallic Materials were presented at the Eighteenth Session of the Fifty-ninth Annual Meeting of the American Society for Testing Materials held in Atlantic City, N. J., June 20, 1956. This Symposium was sponsored by Subcommittee IV on Tension Testing of Committee E-1 on Methods of Testing. A. C. Webber of E. I. duPont de Nemours and Co., Inc., Wilmington, Del., organized the Symposium and presided over the session.

The papers presented were:

Introduction—A. C. Webber
Tension Test Methods for Wood, Wood-Base Materials and Sandwich Constructions—L. J. Markwardt and W. G. Youngquist
Tension Testing of Plastics—Frank W. Reinhart
Tensile Load-Deformation Testing of Textile Structures—Stanley Backer
Tension Testing of Adhesives—A. G. H. Dietz
Tension Testing of Rubber—H. Tangenberg
Present Practices in Tensile Strength Testing of Paper in Industry—John Fachet and F. T. P. Plimpton, Jr.
Summary—A. C. Webber

These papers with discussions with the exception of the paper by Backer, which was not available, were issued as *ASTM Special Technical Publication No. 194* entitled, "Symposium on Tension Testing of Non-Metallic Materials."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON STEAM QUALITY

The introduction of higher pressures and higher steam temperatures in boilers resulted in the vaporization of dissolved solids in the boiler water. Consequently the need for more sensitive and accurate measuring techniques became apparent. This Symposium on Steam Quality reviews the present practices and introduces new techniques for determining minute amounts of solid impurities in steam. It also points up the need for further experimentation in the laboratory and in the field in order to compare the various methods of analysis.

The papers and discussions in this Symposium were presented at the Twenty-sixth Session of the Fifty-ninth Annual Meeting of the American Society for Testing Materials, held in Atlantic City, N. J., June 21, 1956. It was sponsored by the Joint Research Committee on Boiler Feed-water Studies, with M. D. Baker of West Penn Power Co., Springdale, Pa. acting as Symposium chairman and chairman of the technical session.

The papers included in the Symposium are:

Introduction—M. D. Baker
Measurement and Purification of Steam to 0.01 ppm Total Dissolved Solids—W. B. Gurney
Steam Purity Determination by Tracer Techniques—E. E. Coulter and T. M. Campbell, Jr.
Comments on Corrections to Steam Conductivity Measurements—R. O. Parker and R. J. Ziobro
Construction and Operation of Larson-Lane Steam Purity and Condensate Analyzers—A. B. Sisson, F. G. Straub, and R. W. Lane

These papers with discussions were issued as *ASTM Special Technical Publication No. 192*, entitled "Symposium on Steam Quality."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON CORONA

The deleterious effects of corona on organic electrical insulation have been known for years, and considerable research and investigational work has been done by manufacturers and users of organic materials to evaluate their stability in applications where operating voltage gradients approach ionizing levels. However, no common or standard methods of corona detection and evaluation of the corona resistance of insulating materials exist and none have appeared in the literature.

Recognizing the need for standard methods of corona detection and materials evaluation, ASTM Committee D-9 on Electrical Insulating Materials in 1954 organized Section L on Corona, for this purpose. This section immediately launched a program having the following tentative scope: (1) to devise methods to determine the presence of voids in dielectrics which may be ionized under high voltage gradients; and (2) to devise test methods to differentiate relatively among electrical insulating materials with regard to their ability to resist the action of corona and its by-products.

One of the first steps was to organize a symposium on this subject, which was held in November 1955 during the Committee D-9 meeting in Cincinnati, Ohio, with the general purpose of having presented the present test methods and results obtained.

Six of the papers presented, with discussion, are included in this publication:

Introduction—E. B. Curdts
Preliminary Corona Resistance Test Results from IEC Activities—C. W. Ross
Effects of Corona on Plastic Laminates—A. Rufolo and R. R. Winans
Corona Resistance Test Method for Laminates, Molded, and Cast Materials—W. T. Starr
Preliminary Investigation of Proposed Pulse Method for Measuring Ionization—F. S. Oliver, F. H. Povey, and T. A. Pinkham
Corona Detection and Measurement at Sixty Cycles—J. R. Nye
Test Methods for Measuring Energy in a Gas Discharge—S. I. Reynolds

These papers with discussion were issued as *ASTM Special Technical Publication No. 198* entitled "Symposium on Corona."

SUMMARY OF PROCEEDINGS OF SYMPOSIUM ON MINIMUM PROPERTY VALUES OF ELECTRICAL INSULATING MATERIALS

The papers and discussions of this Symposium were presented at a meeting of ASTM Committee D-9 on Electrical Insulating Materials, held in Philadelphia, February 1956.

In the design of electrical equipment the safety factor is based on the minimum expected values for electrical and physical properties of the component materials, as well as an estimate of the extremes of conditions which the equipment may encounter under adverse circumstances. Results of materials tests should enable a prediction as to the lowest expected value for a property of a material so that the designer may take this into account in establishing the safety factor. Unfortunately, this information is not readily obtainable from many present test methods. Though there has been some progress in establishing procedures for predicting minimum values, there is still great need for further work. This symposium may be considered as a report of progress, indicating a baseline for additional investigations toward establishing safety factor on a basis of knowledge rather than ignorance.

The papers presented were:

Significance of Minimum Property Values of Electrical Insulating Materials —C. L. Craig

Extremal Nature of Dielectric Breakdown—Effect of Sample Size—H. S. Endicott and K. H. Weber

Variability of Dielectric Breakdown in Sheet Insulation—H. K. Graves

The Establishment of Minimum Quality Levels of Sheet Insulation—C. J. Photiadis

Low-Temperature Resistance Test Methods for Non-Rigid Insulation—C. L. Craig

Statistical Treatment of Heat-Aging Studies on Varnished Glass Cloth—L. E. Sieffert and E. W. Alexander

Quality Level Cannot Be Determined by "Go"—"No Go" Gage—L. J. Timm

These papers with discussions were issued as *ASTM Special Technical Publication No. 188*, entitled "Symposium on Minimum Property Values of Electrical Insulating Materials."

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LIST OF SPECIAL TECHNICAL PUBLICATIONS

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7-B	Significance of Tests of Petroleum Products (1956)
56-T	1955 Supplement to the Bibliography and Abstracts on Electrical Contacts (1956).
58-C	Report on Standard Samples and Related Materials for Spectrochemical Analysis, Compiled by Robert E. Michaelis (1956).
150-A	Bibliographical Abstracts for Methods for Analysis of Synthetic Detergents (1888–1956)
186	Method for Reducing the Effect of Barometric Pressure in Measurement of Octane Number (1956)
187	Relaxation Properties of Steels and Super-Strength Alloys at Elevated Temperatures (1956)
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213	*Symposium on Nondestructive Testing (1956)
214	*Symposium on Railroad Materials Including Lubricating Oils (1956)
215	*Symposium on Radioactive Isotopes (1956)

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*West Coast Papers.